Liquid crystals in nanometric confinement

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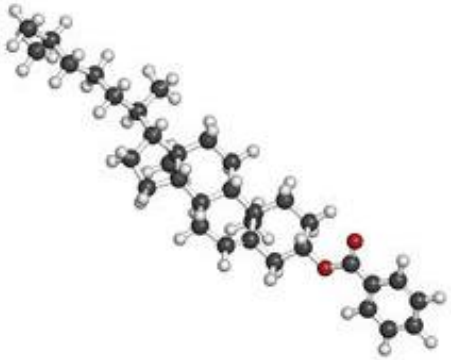
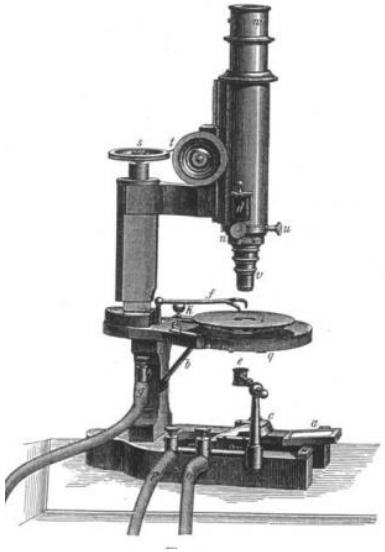
Discovery of liquid crystals (LCs)

Friedrich Reinitzer



Cholesteryl benzoate

1888 r.



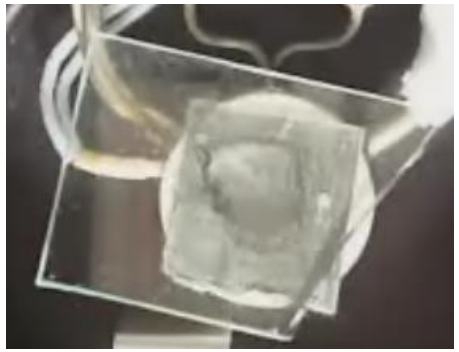
Otto Lehman

Cr

145 °C

LC

178.5 °C

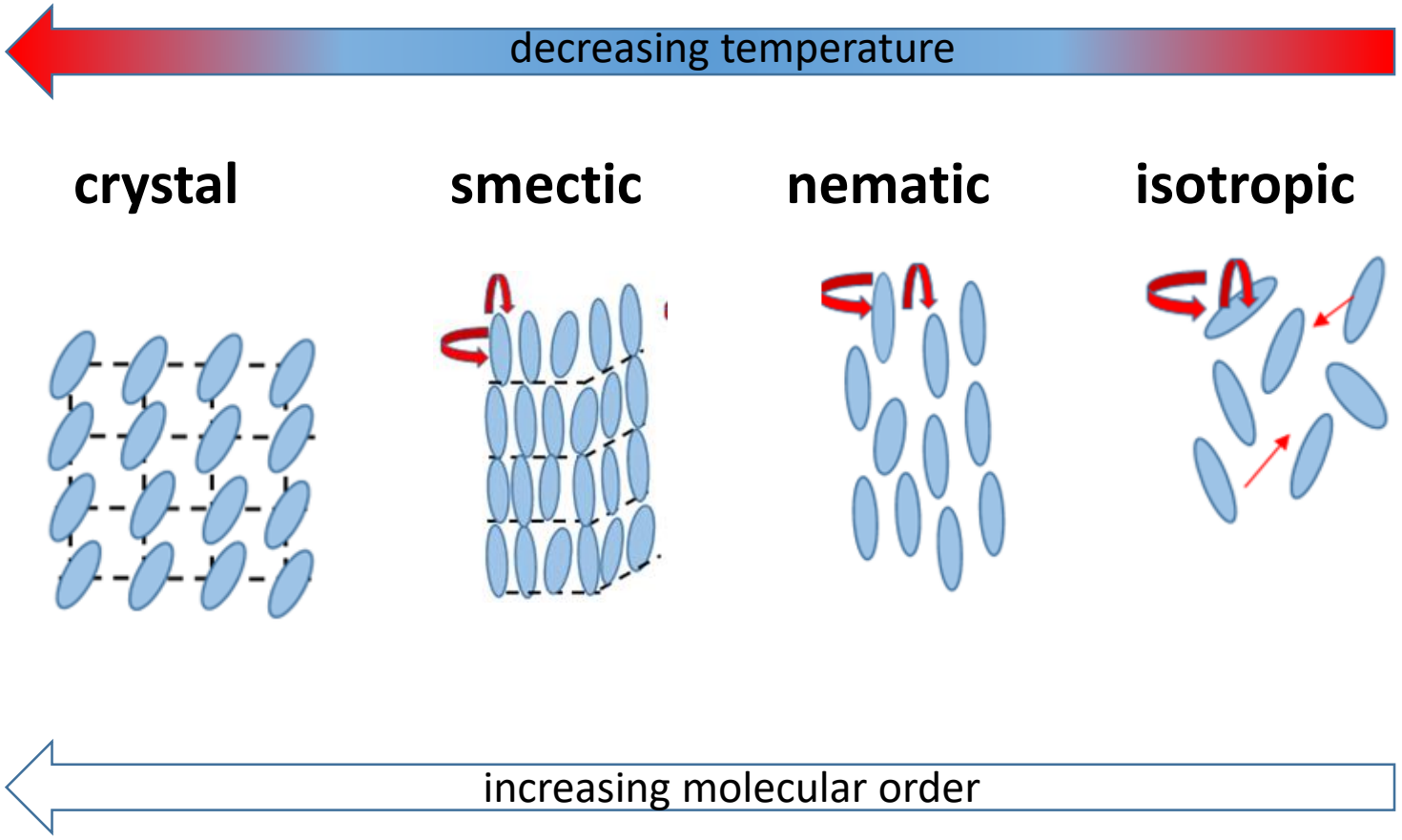


I

From a letter to Reinitzer:

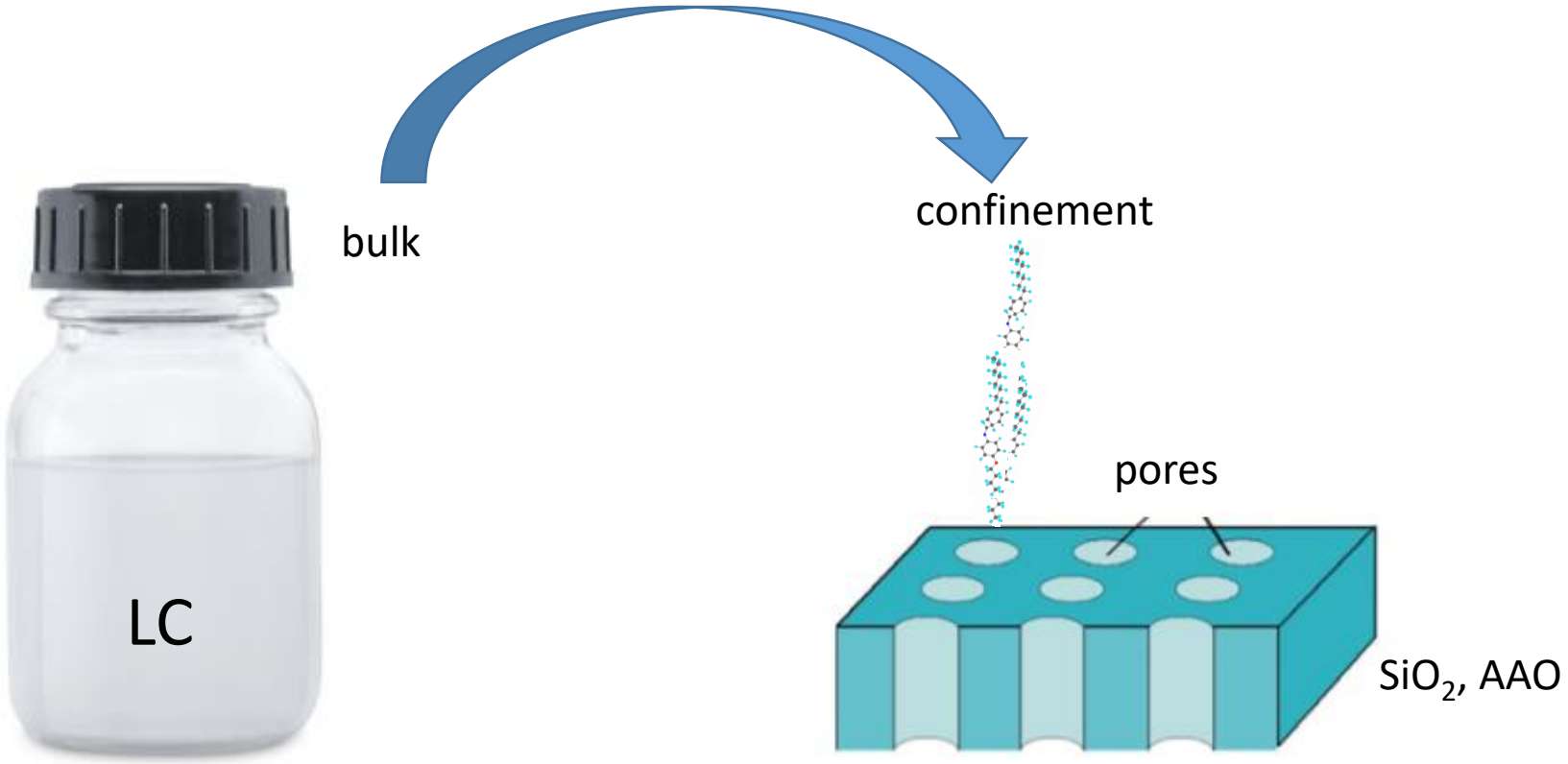
"... my new results confirm your [previously] declared view, that the [substance] consists of very soft crystals ... It is absolutely homogeneous, and another liquid - as you assumed formerly - is not present ... It is of a high interest for the physicist that crystals exist which are of such a considerable softness that one could almost call them liquid."

Possible phase sequence in liquid crystals



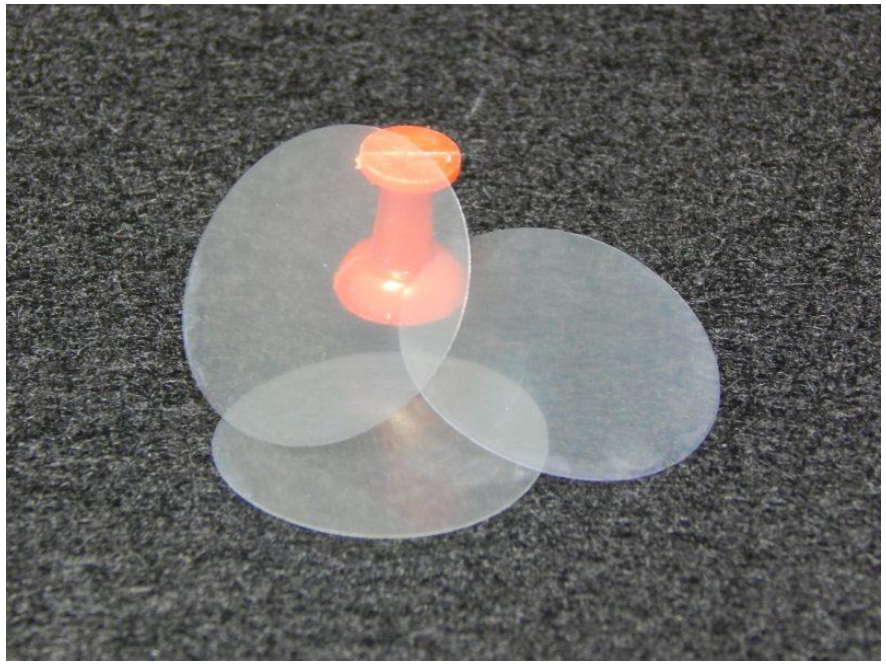
As the temperature decreases, a LC compound forms phases of higher degree of order.

Motivation

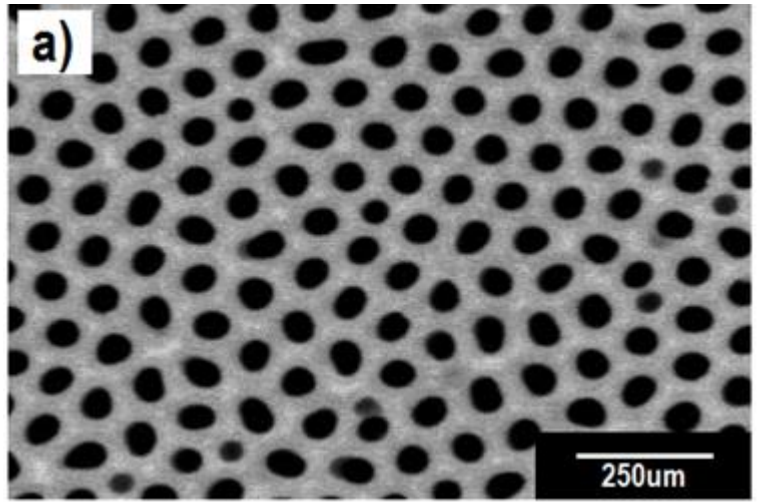


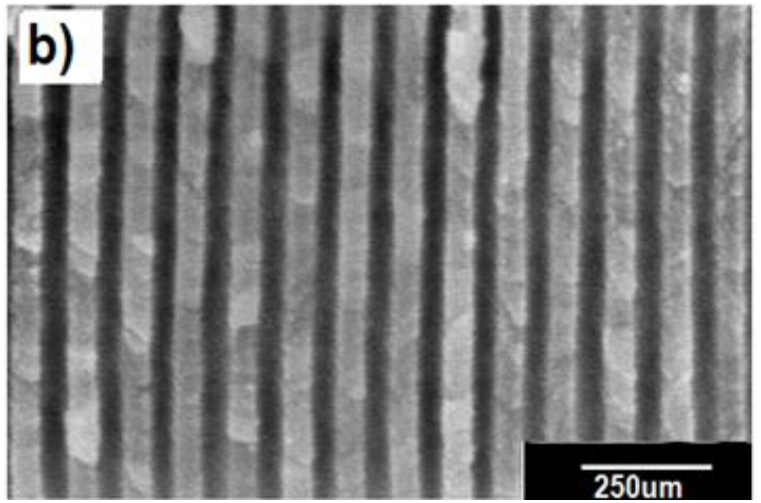
The main objective of our research was to understand better the impact of confinement on phase sequence and molecular motions in liquid crystals. Control of phase behaviour and ordering of molecules through spatial constraints will allow to design materials with adjustable optical, dielectric properties.

Behaviour of LCs in pores with diameter of several dozen nanometers

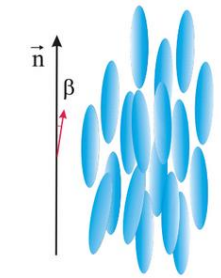


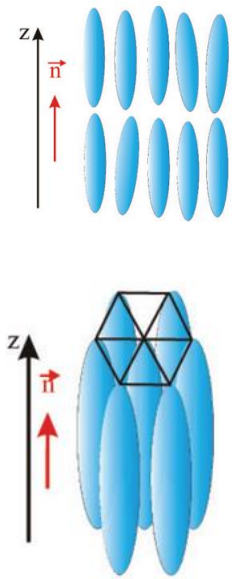
Nanoporous Anodic Aluminum Oxide (AAO)

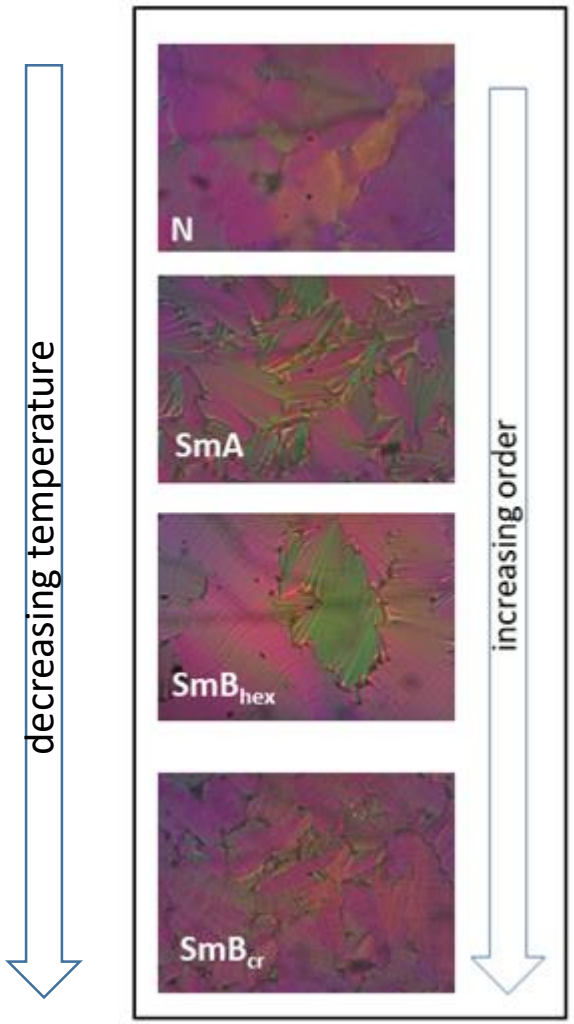


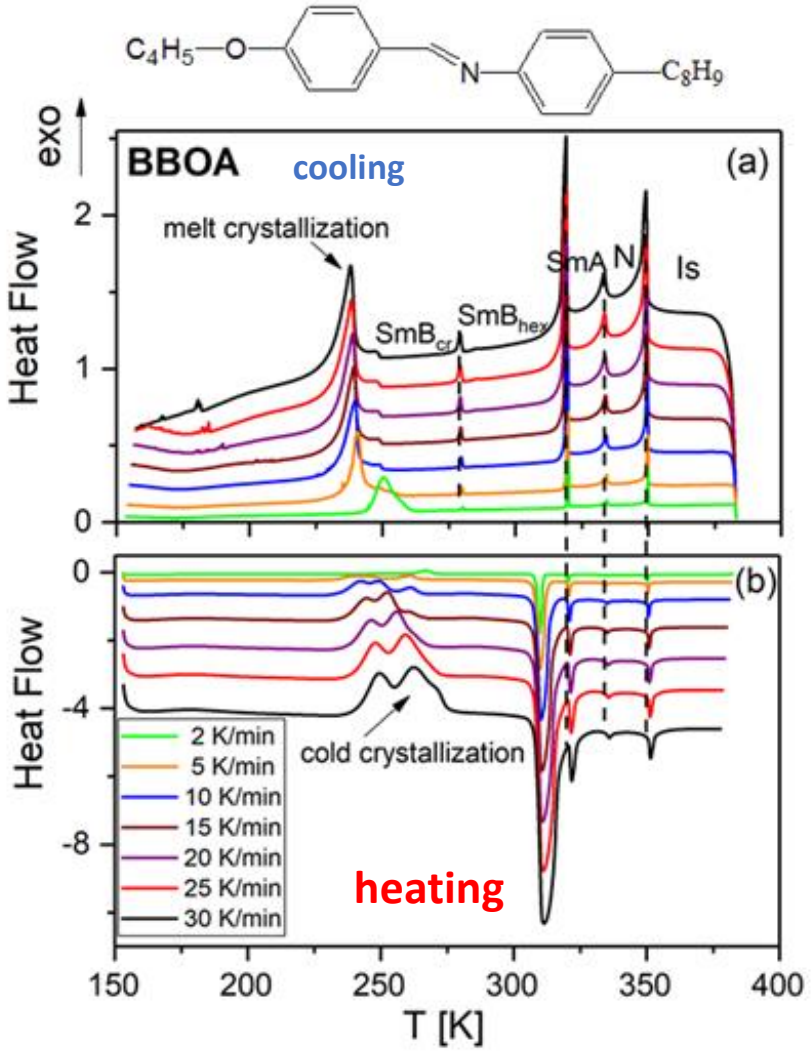


Phase sequence of BBOA in bulk









enthalpy

$$\Delta H = \int_{T_0}^{T_\infty} \frac{\Delta Q}{\Delta t}\frac{\Delta t}{\Delta T} dT$$

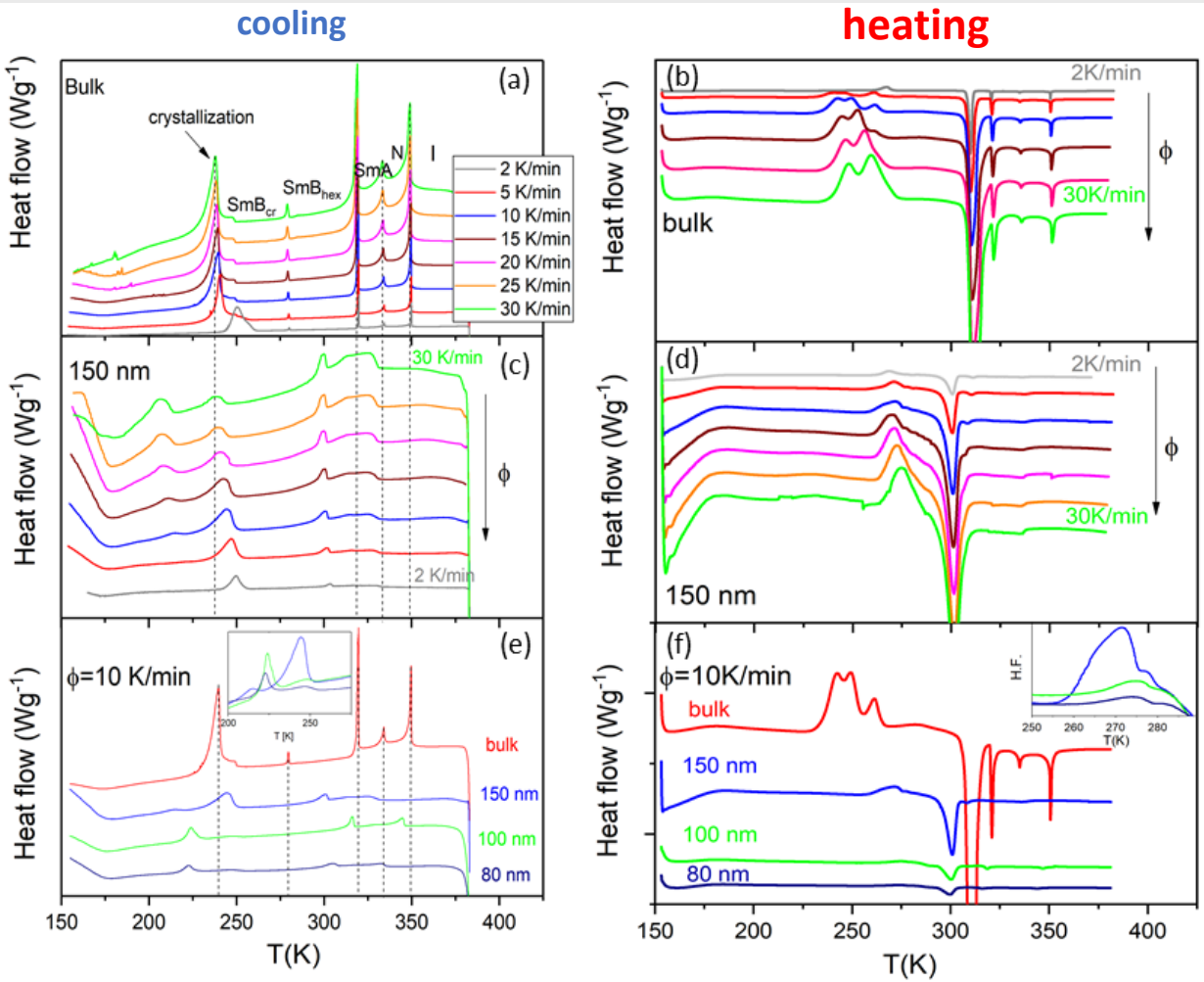
$$\Delta S = \frac{\Delta H}{T_p}$$

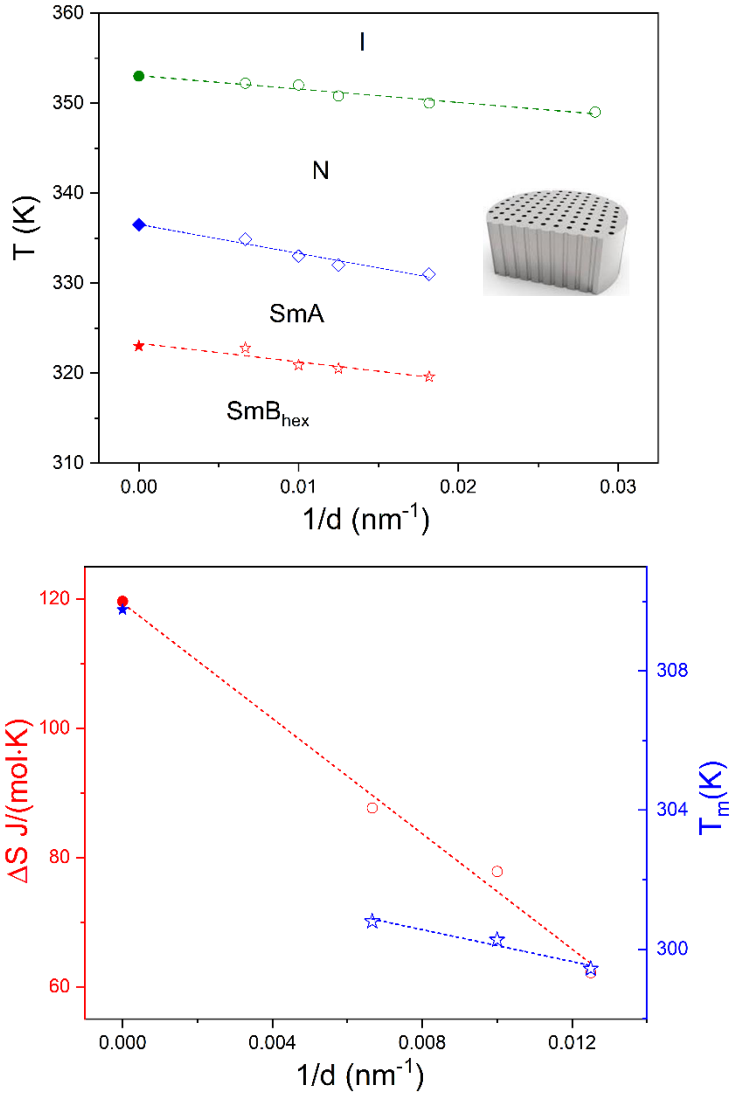
entropy

DSC (Differential Scanning Calorimetry) thermograms

M. Jasiurkowska-Delaporte, T. Rozwadowski, E. Juszyńska-Gałązka Crystals, 9 (2019) 205

Impact of confinement on phase transitions of BBOA

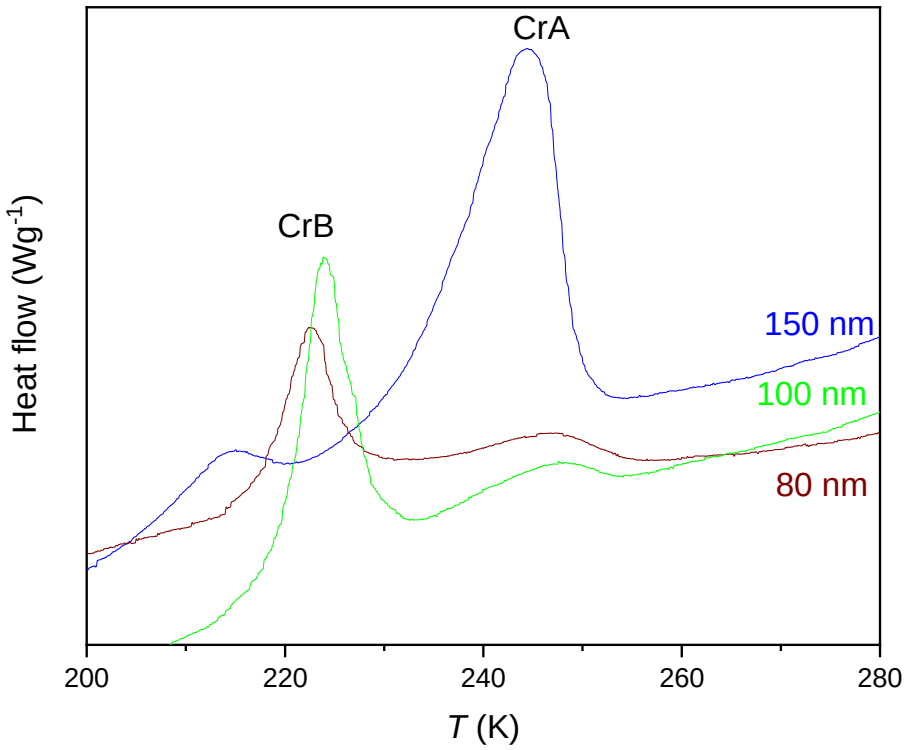




The temperatures of the I-N, N-SmA, SmA-SmB_{hex} transitions are lineary reduced with the reciprocal pore diameter.

***M. Jasiurkowska-Delaporte**, E. Juszyńska-Gałązka, P. M. Zieliński, M. Marzec,J. Mol. Liq., 308 (2020) 113039.*

Non-isothermal crystallization process of BBOA in pores



The formation of CrA is favored in 150 nm pores whereas confinement of BBOA in smaller pores (100 nm and 80 nm) facilitates crystallization of CrB phase.

M. Jasiurkowska-Delaporte, E. Juszyńska-Gałązka, P. M. Zieliński, M. Marzec,J. Mol. Liq., 308 (2020) 113039.

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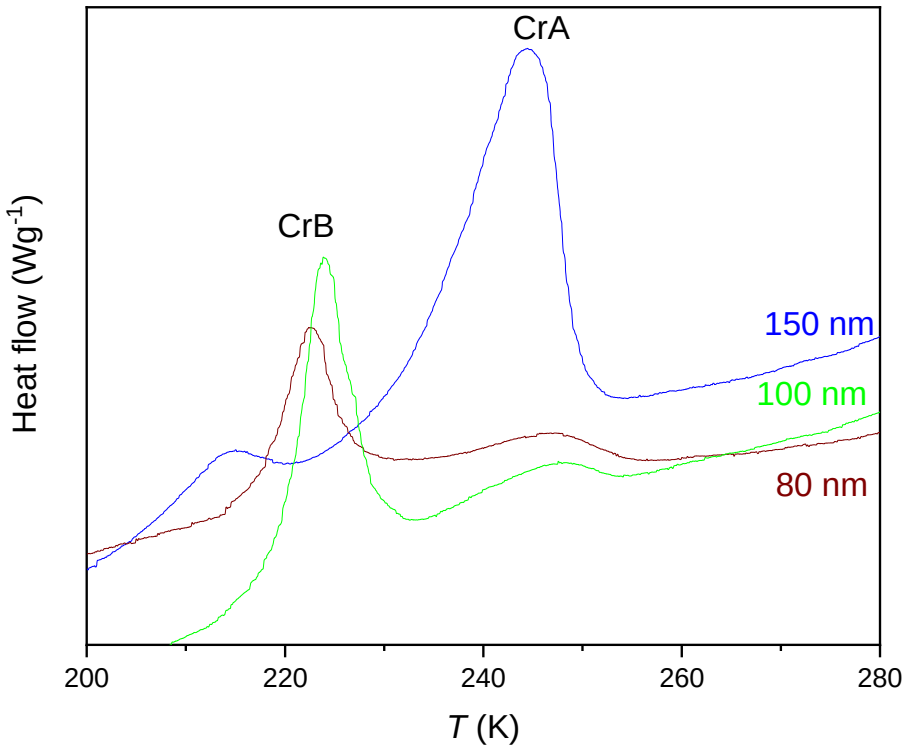

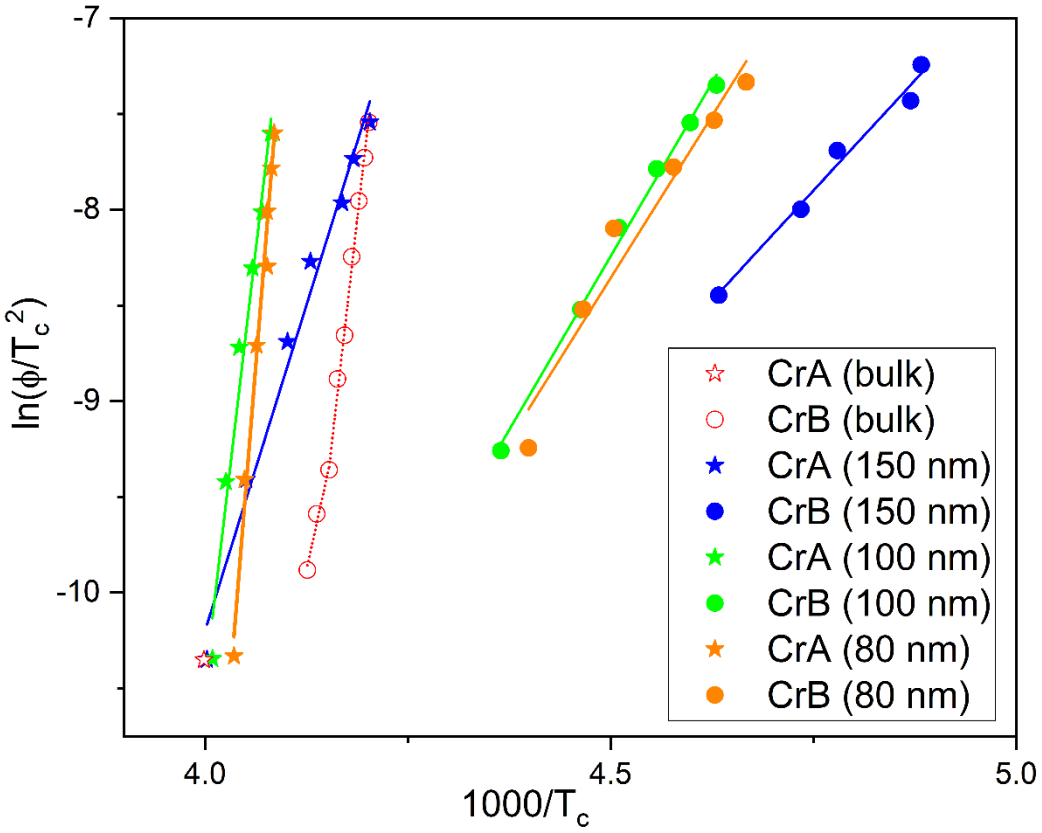

The activation barrier of crystallization of CrA is a few times higher than that one determined for formation of CrB.

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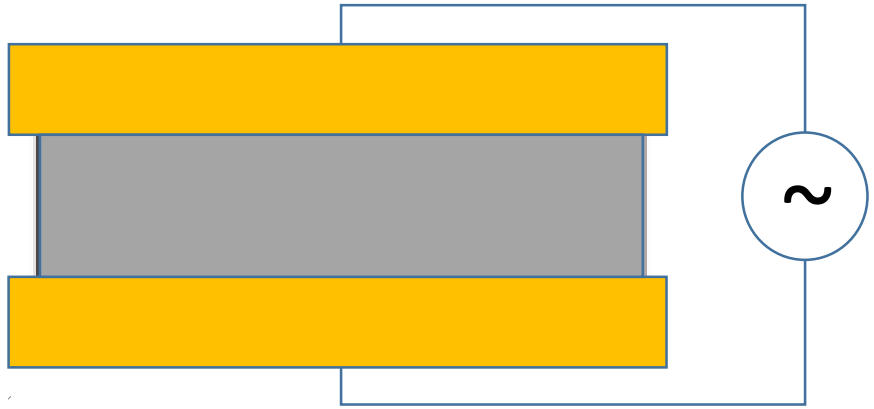
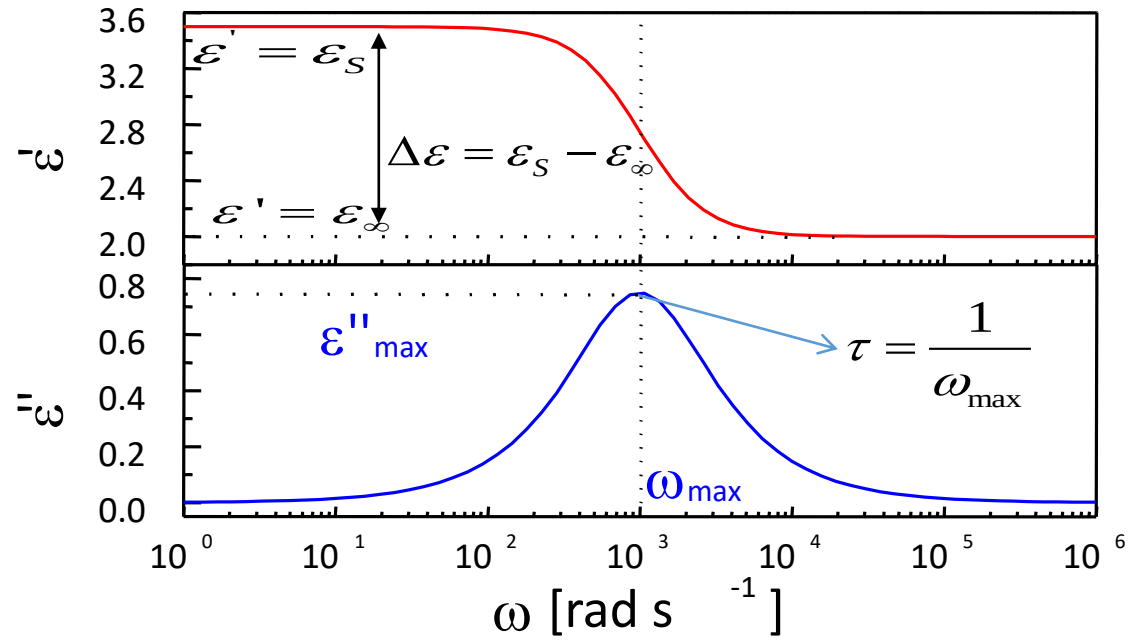
Broadband dielectric spectroscopy (BDS)

BDS- measures complex dielectric function $\varepsilon^*(\omega) = \varepsilon'(\omega) - i\varepsilon''(\omega)$ in a wide temperature and frequency range.

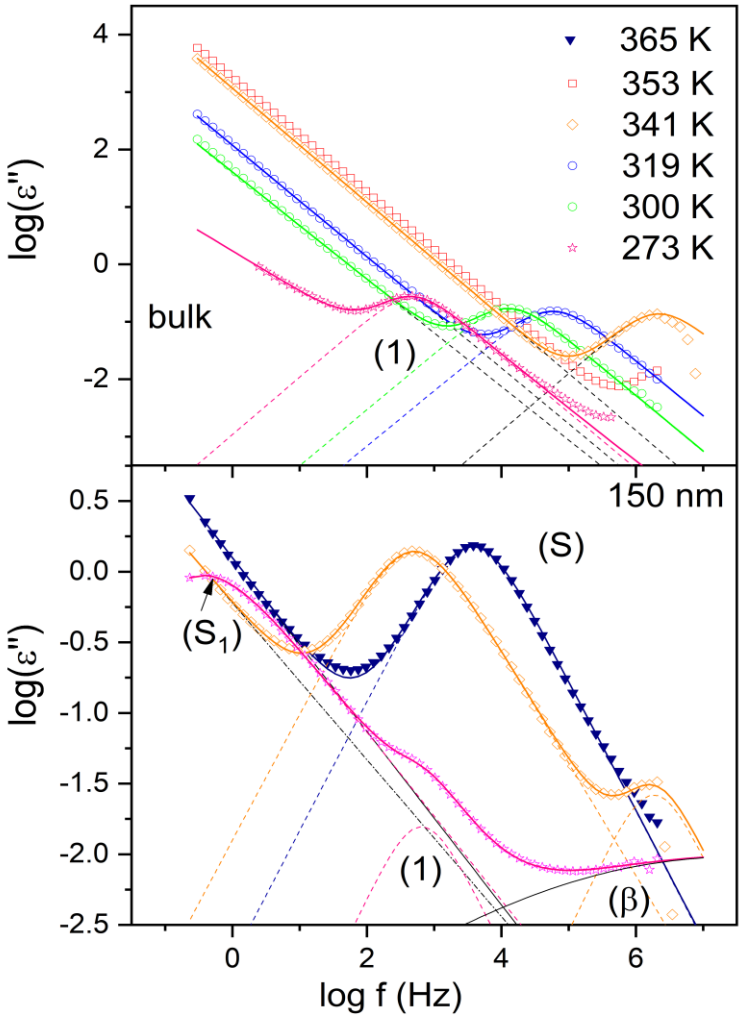


$U_t = U_0 \sin(\omega t)$

$$\varepsilon^*_{HN}(\omega) = \varepsilon_\infty + \frac{\Delta\varepsilon}{(1+(i\omega\tau_{HN})^\beta)^\gamma}$$



Relaxation processes of BBOA in pores



(S) – process

Maxwell - Wagner (MW) polarization process

or

paranematic state (PN)

(A.V. Kity et al., PRL 2008)

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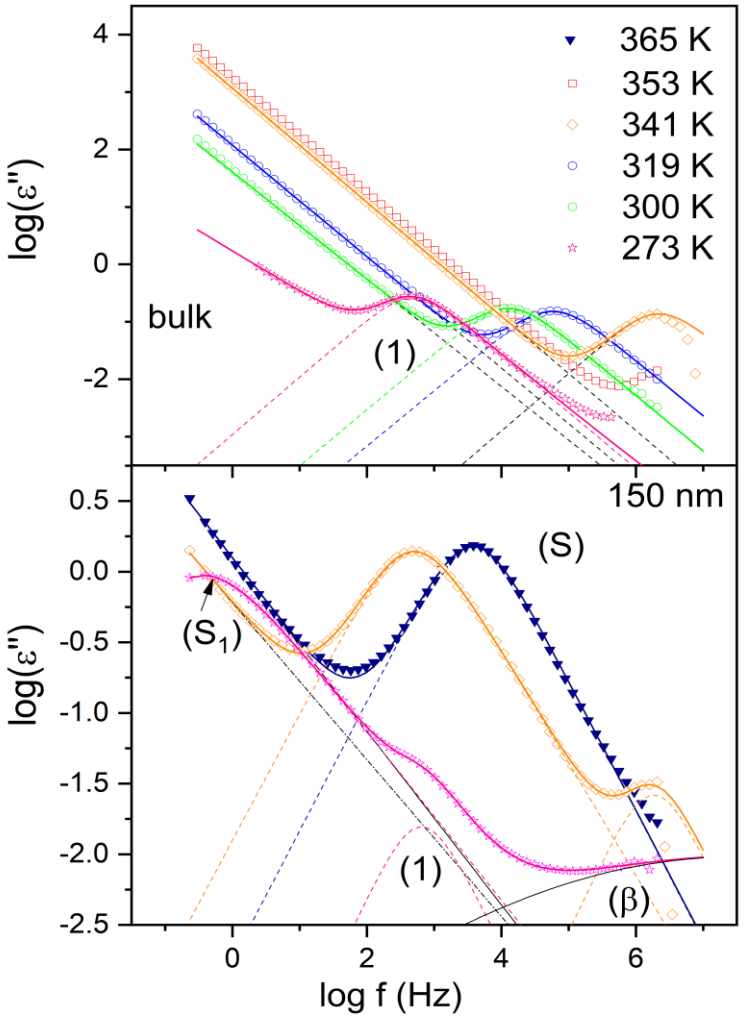

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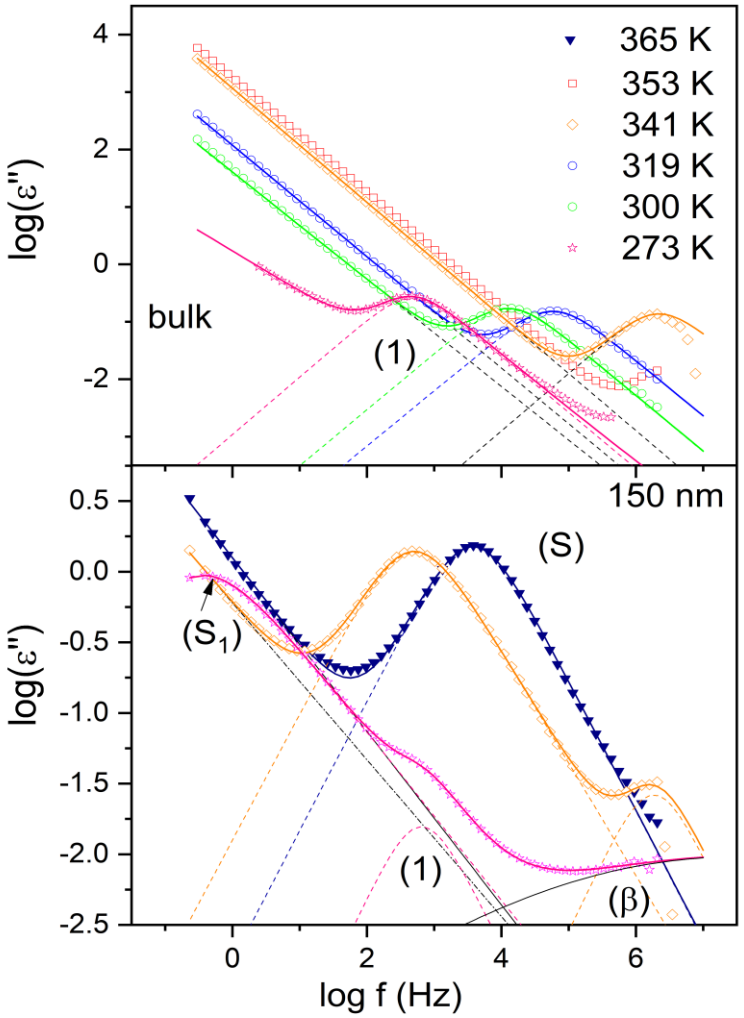
$$\tau_{MW} = 2\pi\varepsilon_0 \frac{2\varepsilon_{AAO} + \varepsilon_{LC} + \varphi(\varepsilon_{AAO} - \varepsilon_{LC})}{\sigma(1-\varphi)}$$

$\varepsilon_{Al_2O_3}, \varepsilon_{LC}$- the dielectric permittivity of AAO matrix and a LC, respectively

σ - the conductivity of LC

φ - a volume fraction of pores.

Relaxation processes of BBOA in pores



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The estimated relaxation time τ_{MW} is much longer than the experimentally determined value of $\tau_{(S)}$.

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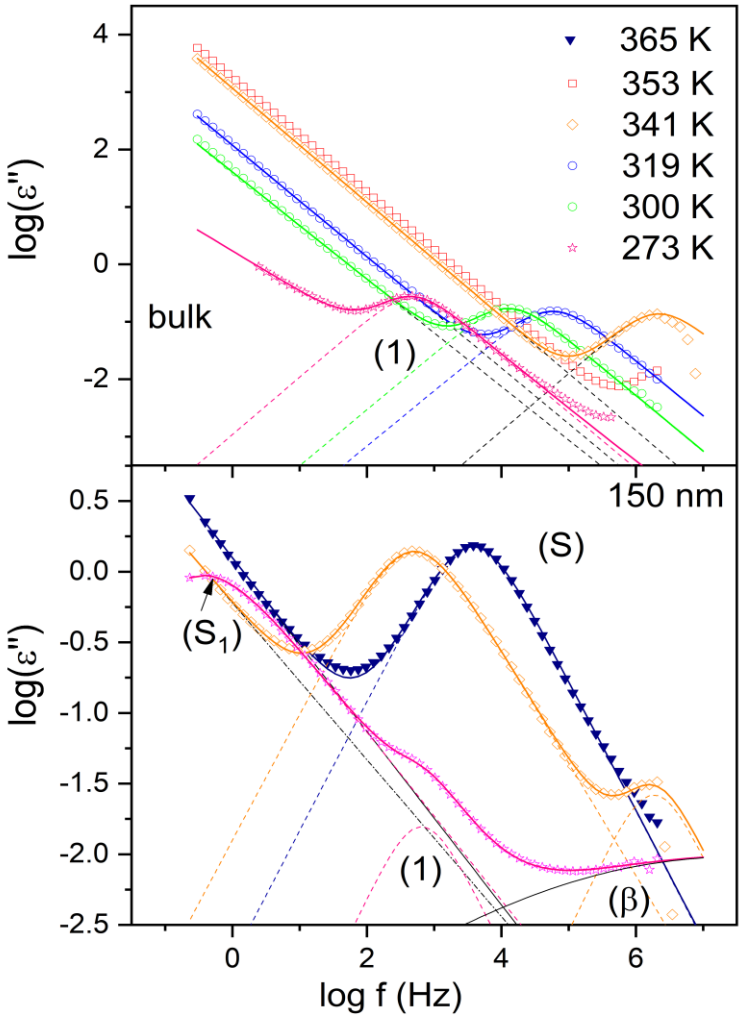

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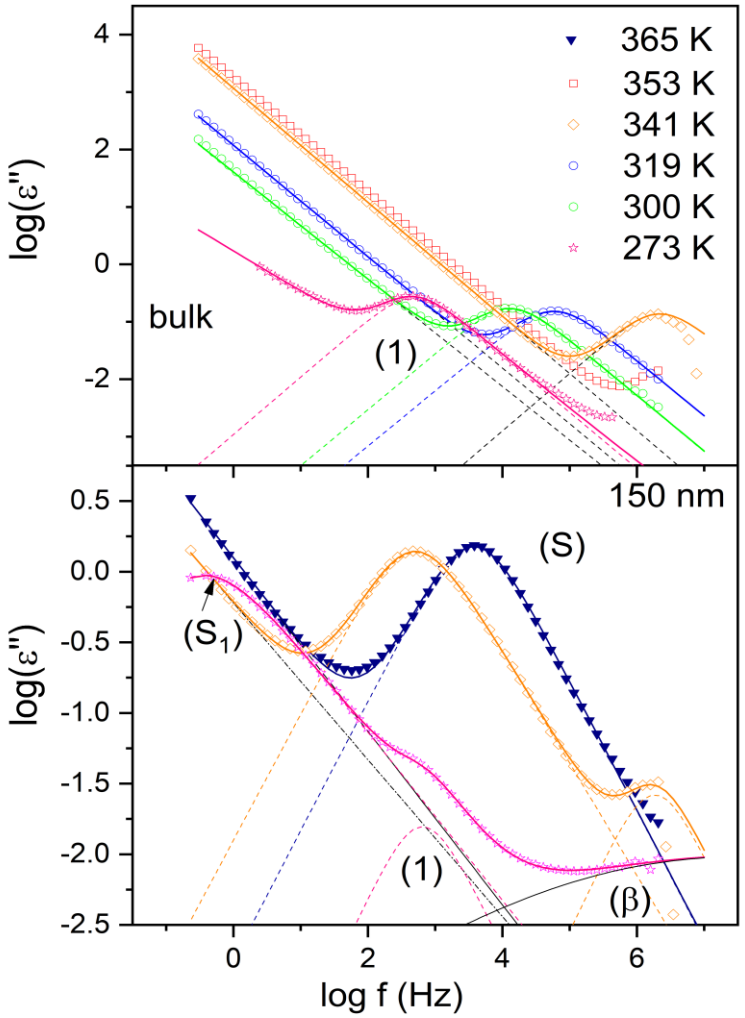
The (S) process is associated with PN state.

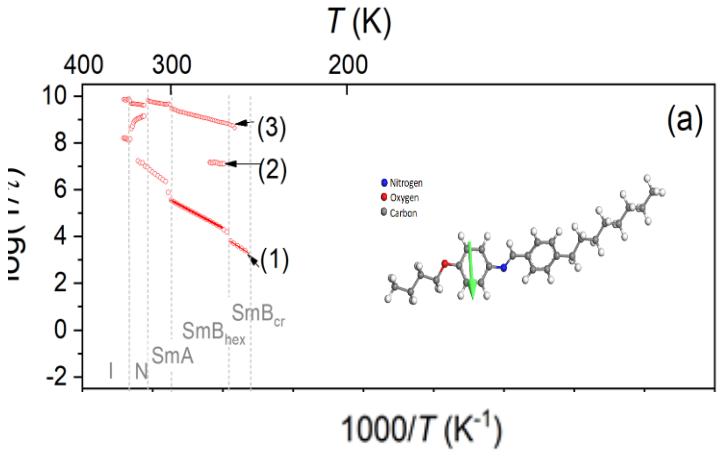
M. Jasiurkowska-Delaporte, E. Juszyńska-Gałązka, P. M. Zieliński, M. Marzec,J. Mol. Liq., 308 (2020) 113039.

Relaxation processes of BBOA in pores



Arrhenius equation

$$\tau = \tau_{\infty} \exp\left(\frac{E_a}{RT}\right)$$



New relaxation process associated with paranematic state was revealed in BDS studies.

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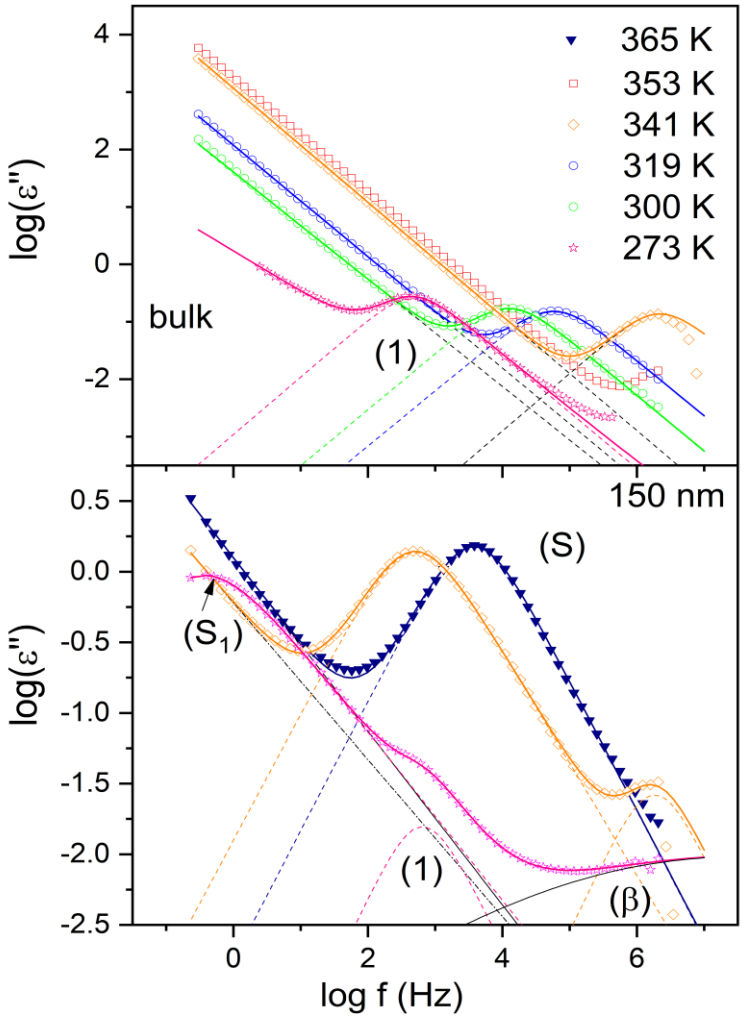
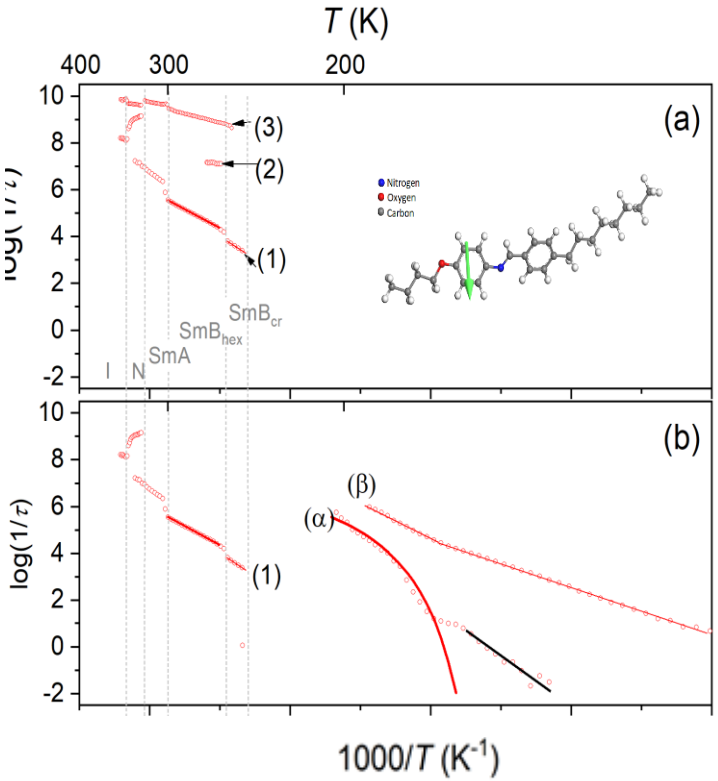


Arrhenius equation

$$\tau = \tau_\infty \exp\left(\frac{E_a}{RT}\right)$$

Vogel–Fulcher–Tammann (VFT) equation

$$\tau_\alpha = \tau_\infty exp\left(\frac{D_f T_0}{T - T_0}\right)$$



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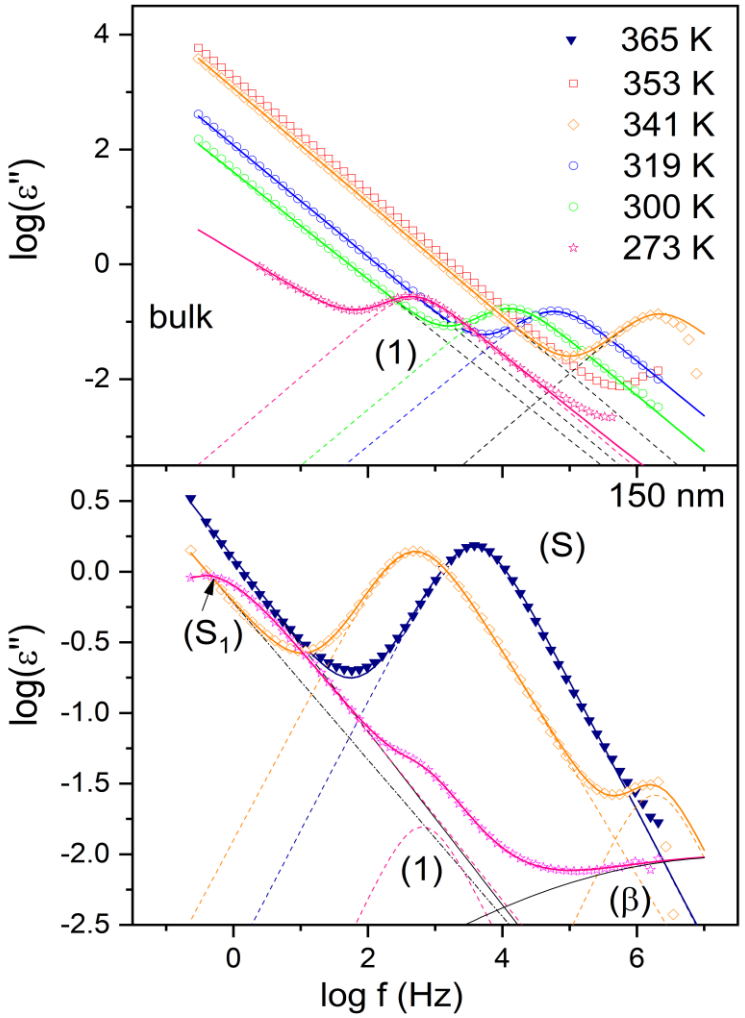
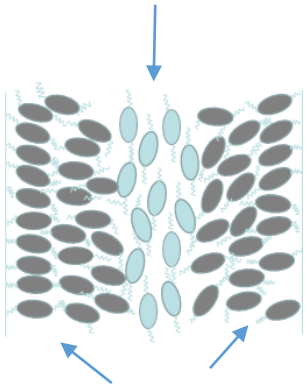
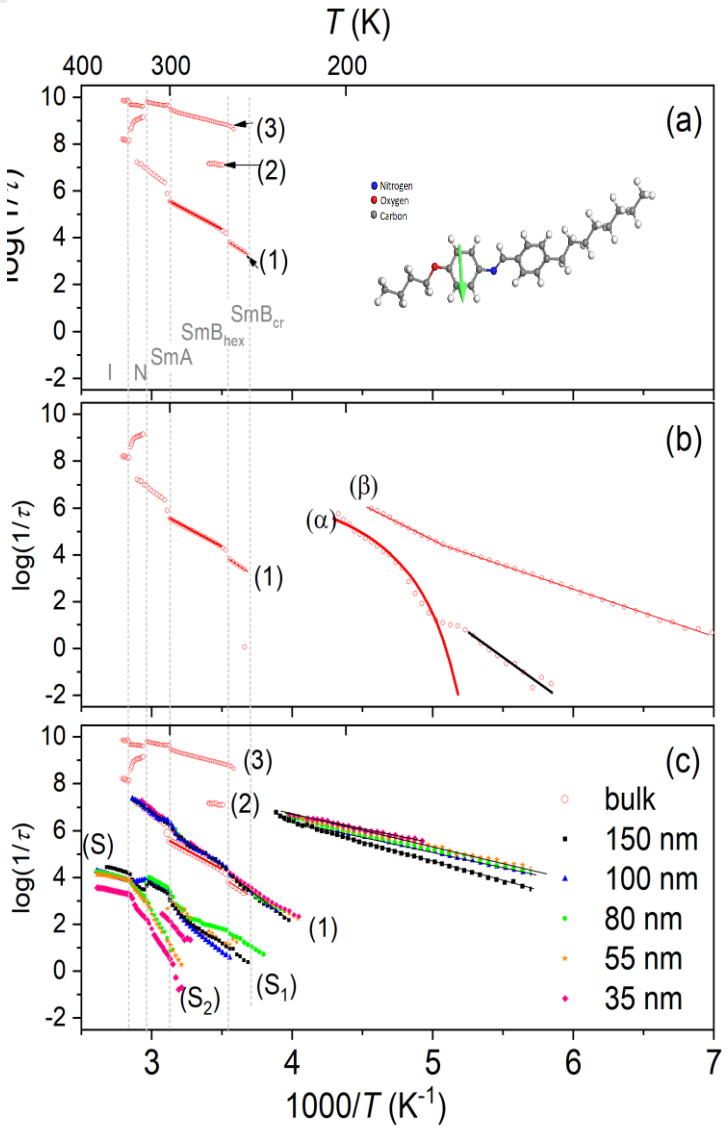


volume molecules

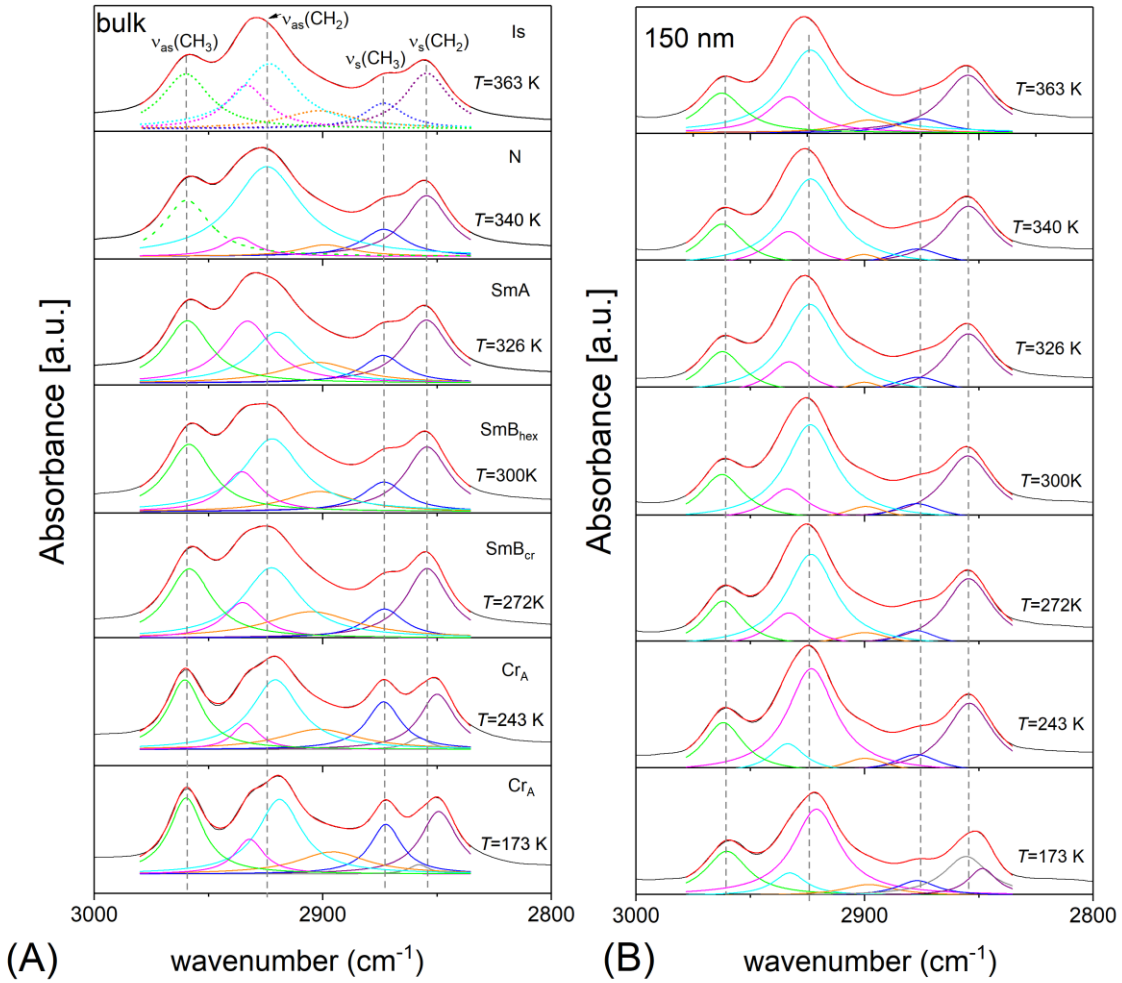


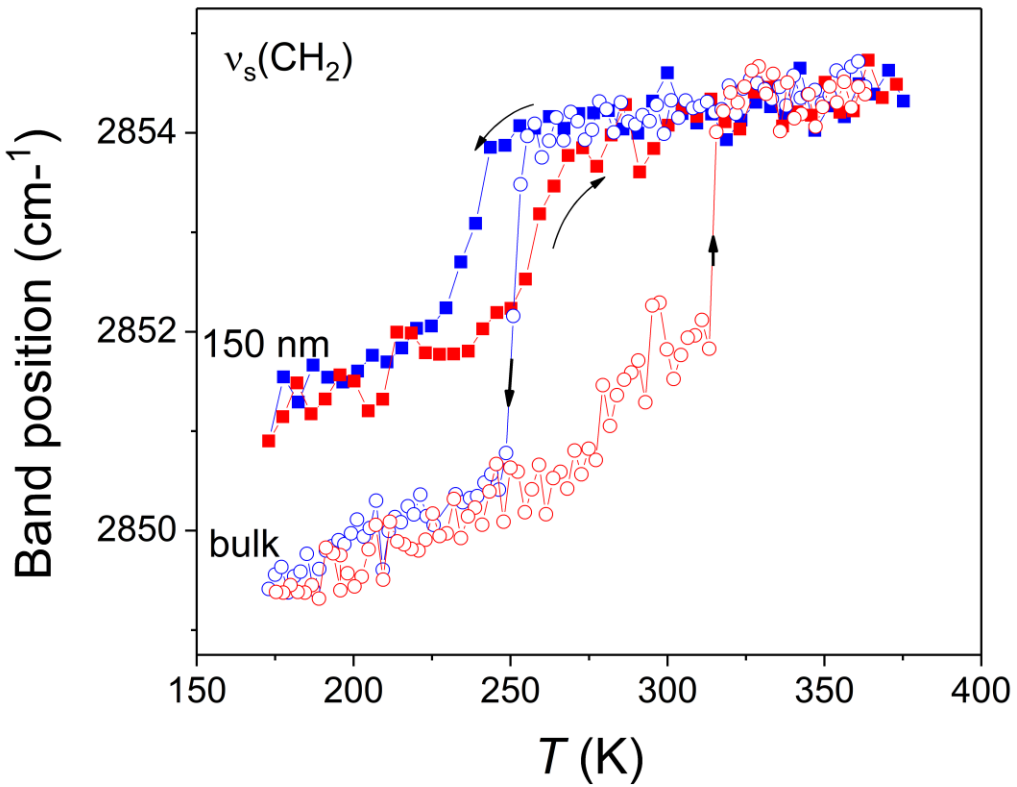
Surface layer



New relaxation process associated with paranematic state was revealed in BDS studies.

The influence of nanoconfinement on intramolecular dynamics





The gradual crystallization process in pores is also reflected in vibrational dynamics of alkyl chains.

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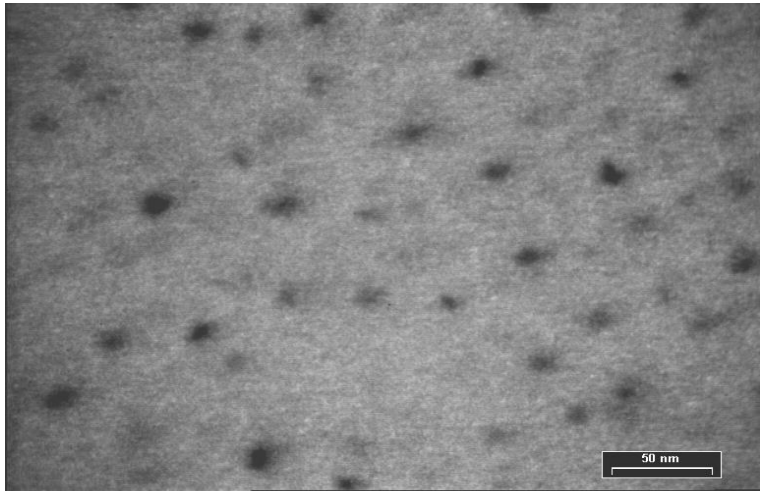
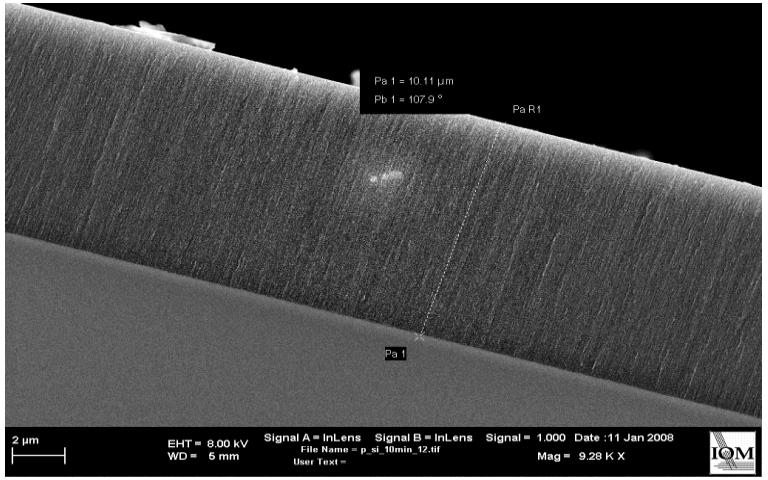
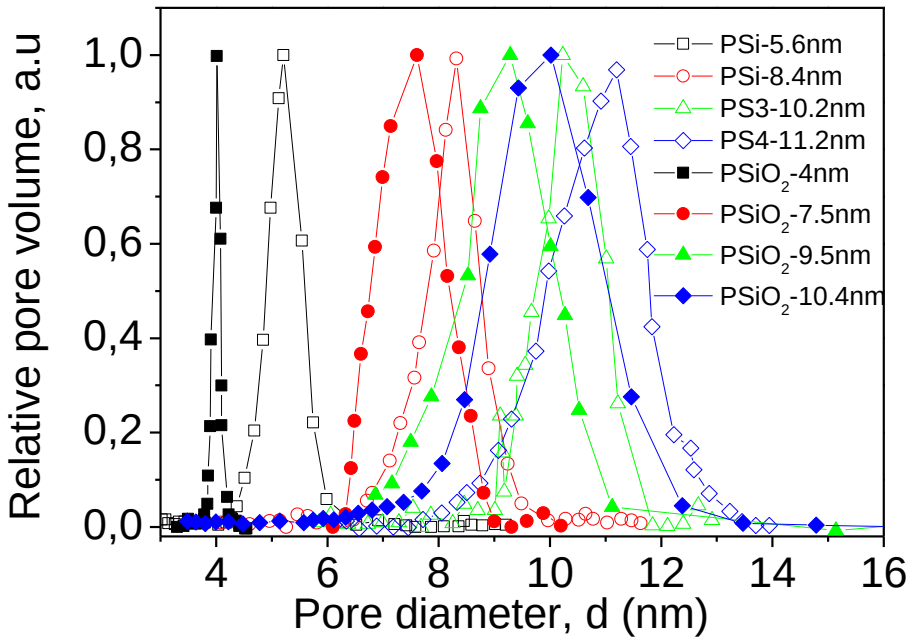
LC behaviour in pores of a few nanometer size



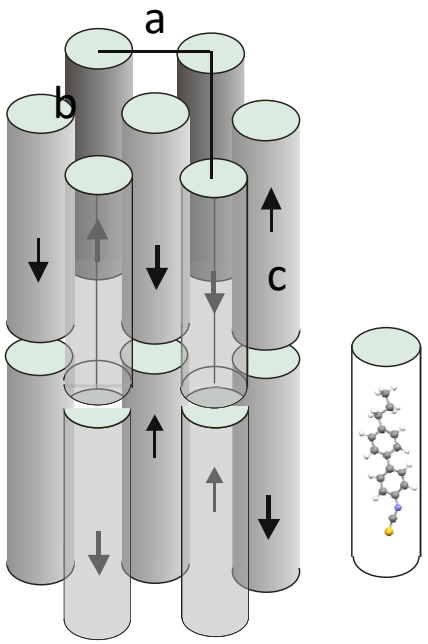




Silica porous membranes (SiO_2)

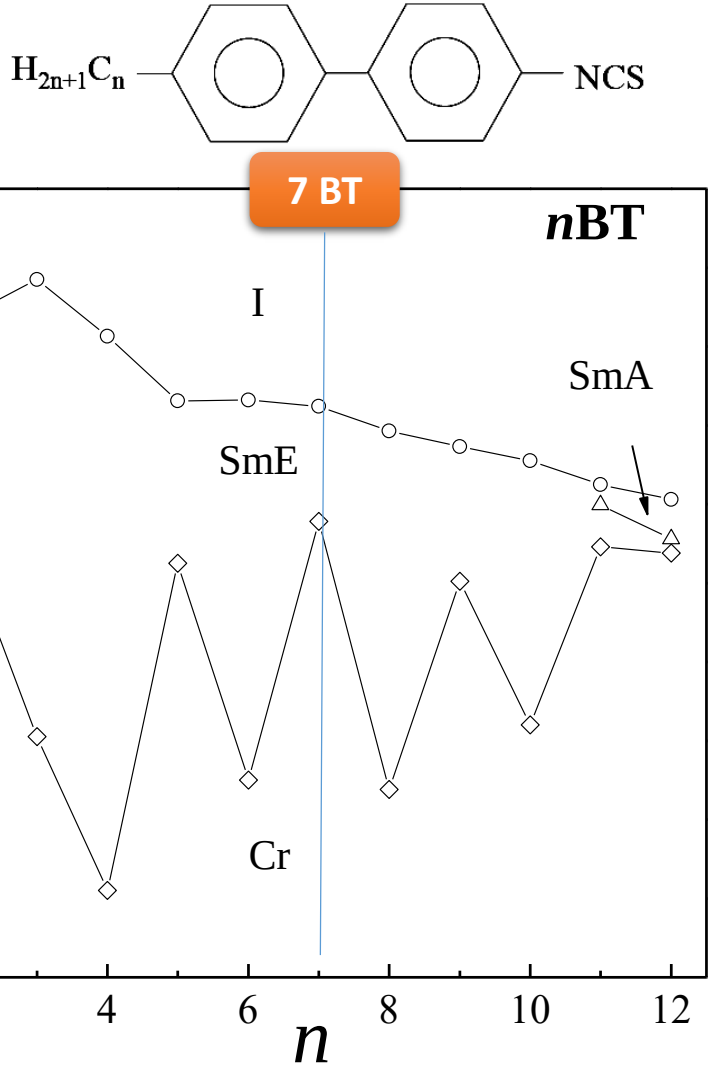
Investigated liquid crystalline compound

Smectic E



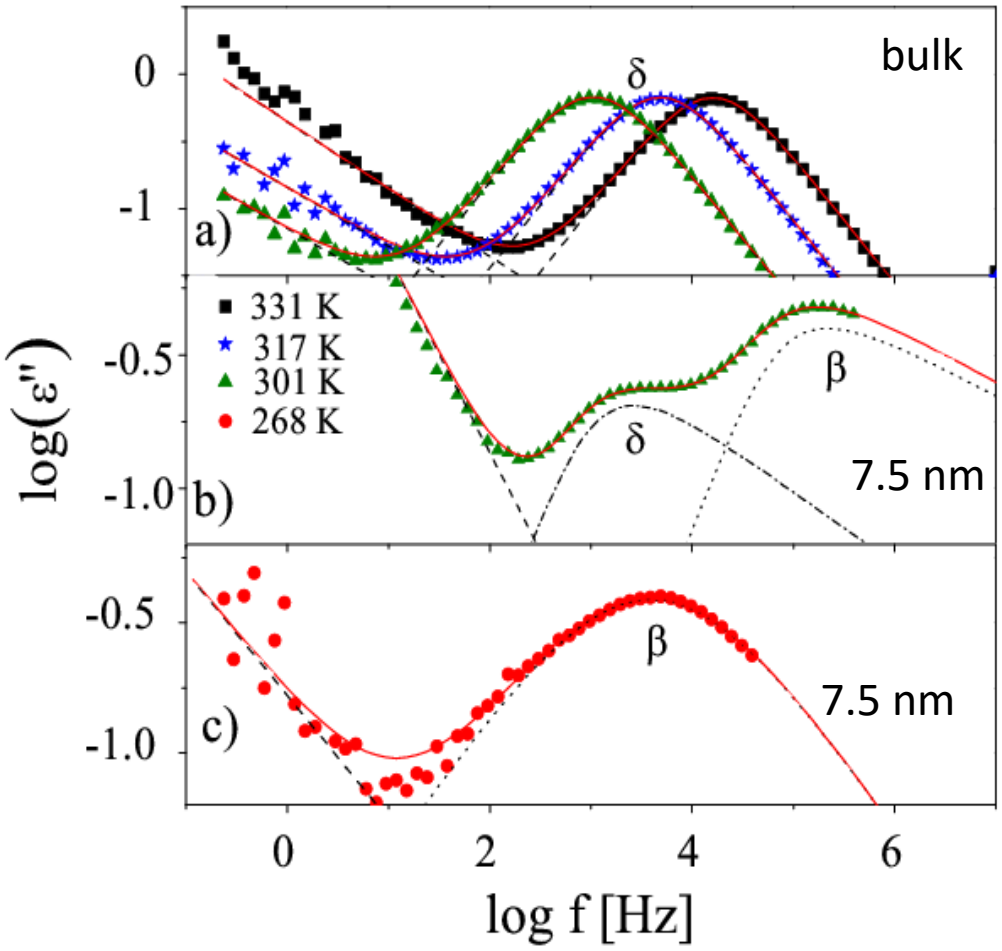
a=0.8 nm
b= 0.5 nm
c=0.2 nm

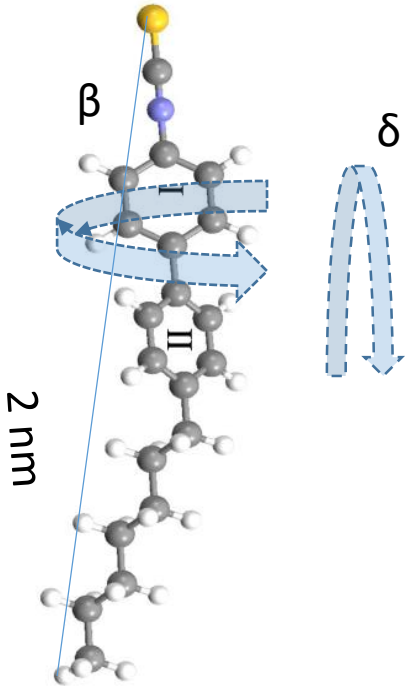
a – distance between the center of mass of two neighbouring molecules
b – diameter of the molecules
c – layer thickness



6BT bulk shows one mesophase: smectic E (SmE) phase.

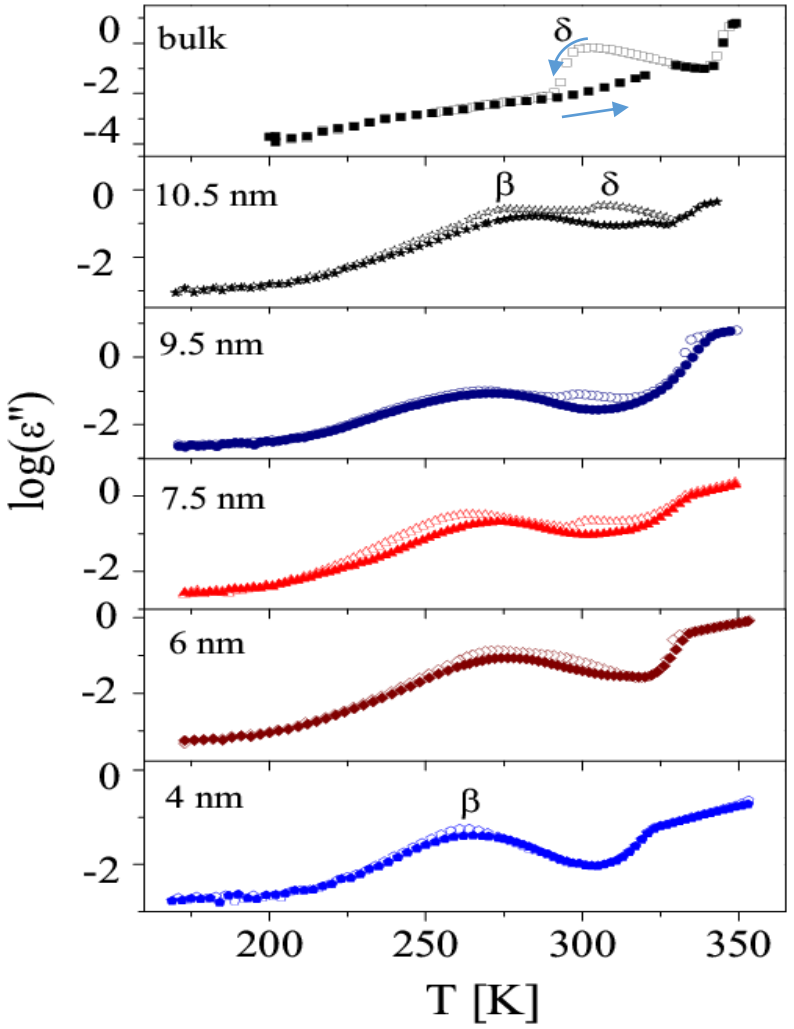
Relaxation processes of 7BT in bulk and confined to 7.5 nm pores

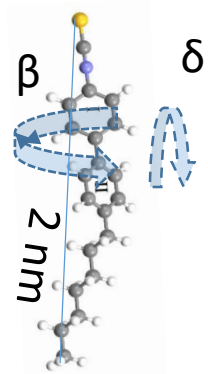


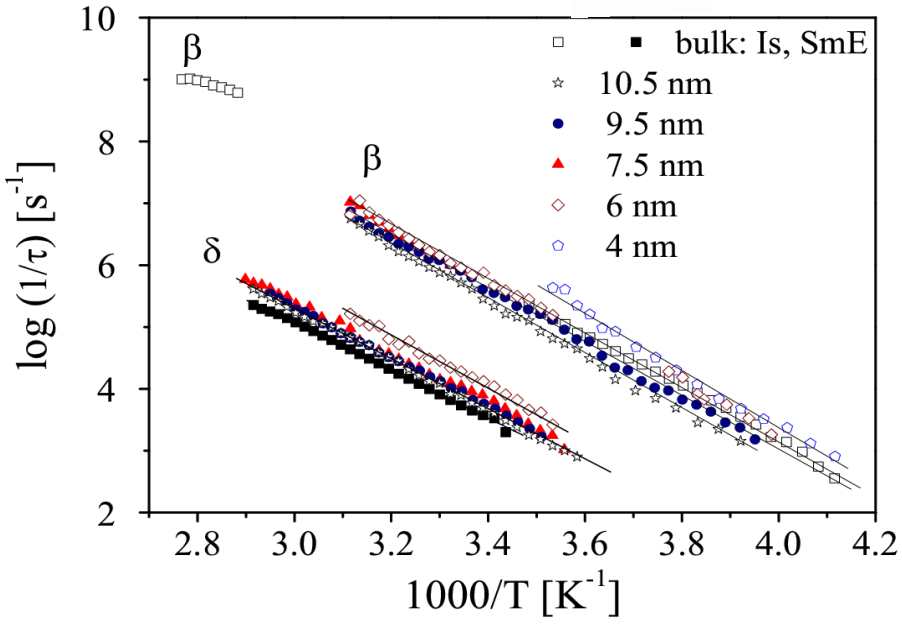


In nano-pores an additional process attributed to libration motions of molecules close to pore walls is detected.

The impact of pore size on molecular dynamics of 7BT LC

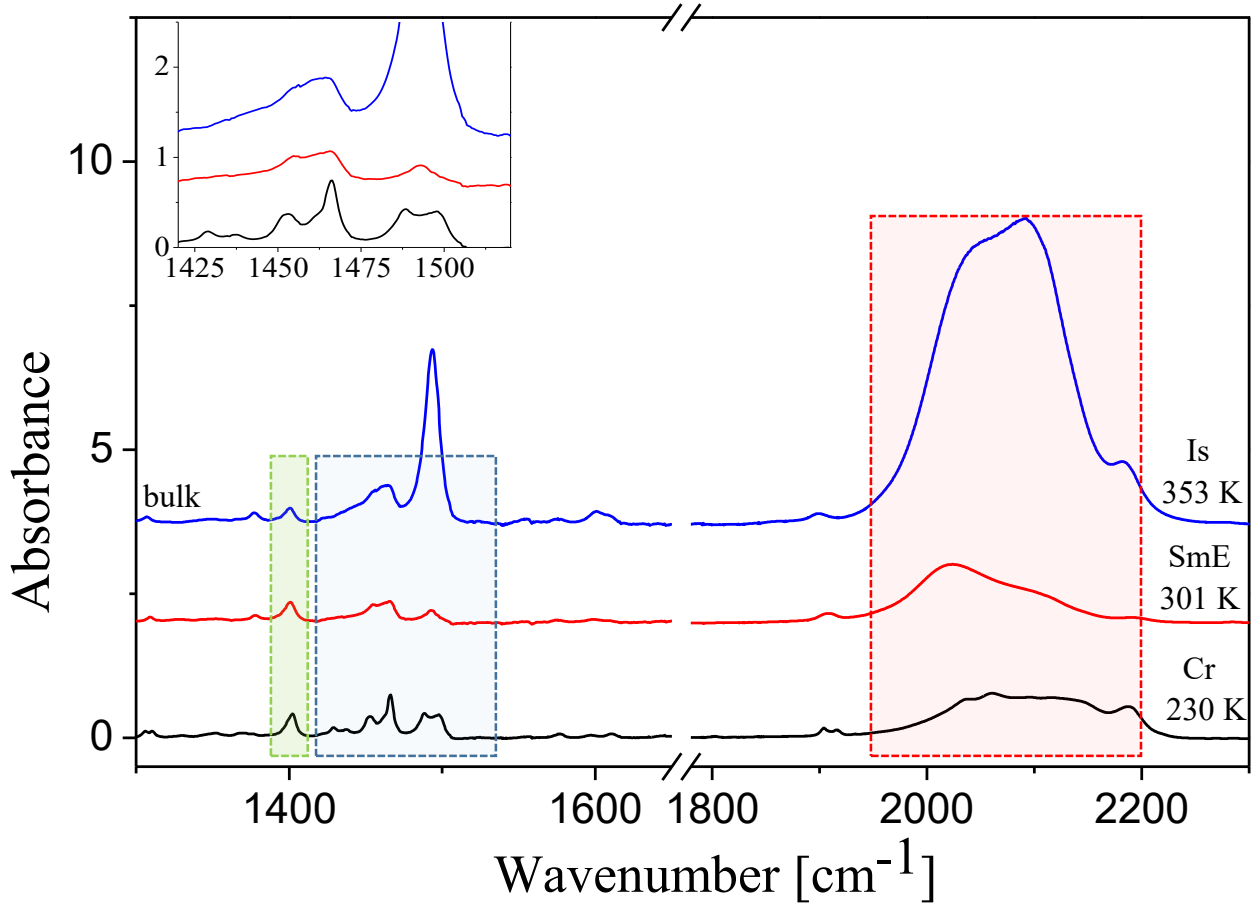


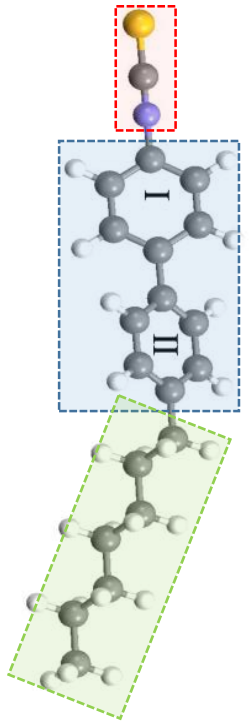




As the pore size decreases the surface effect becomes more pronounced and the δ – relaxation is suppressed.

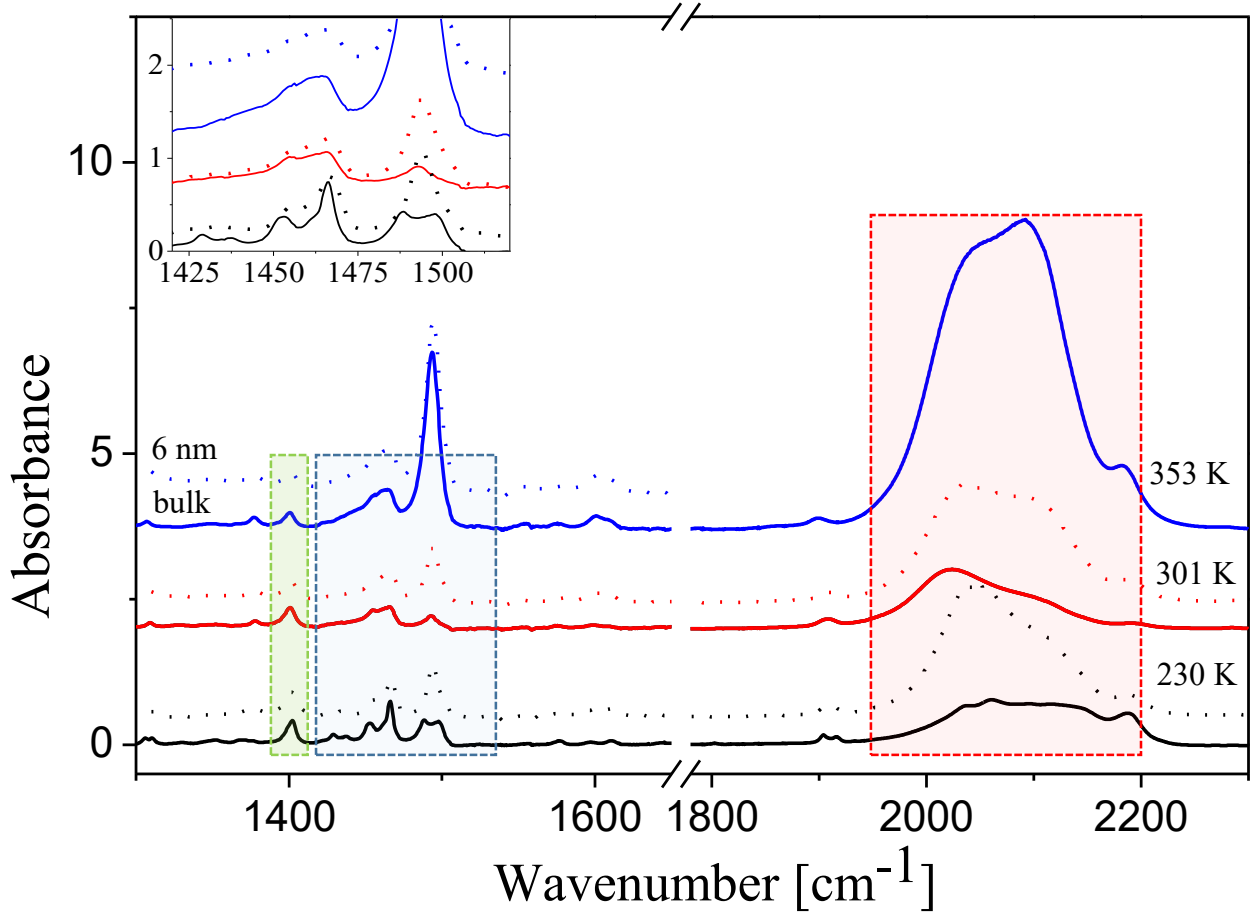
Vibrational dynamics of 7BT in bulk

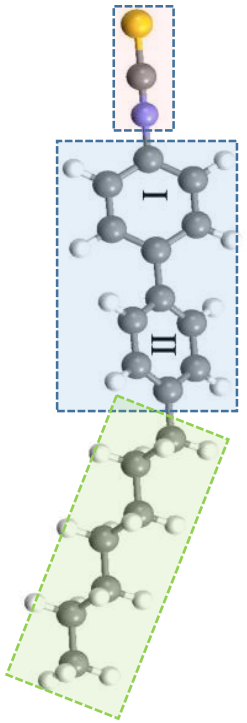




Significant deferences in IR spectra are observed for the isotropic, SmE and crystalline phases in bulk.

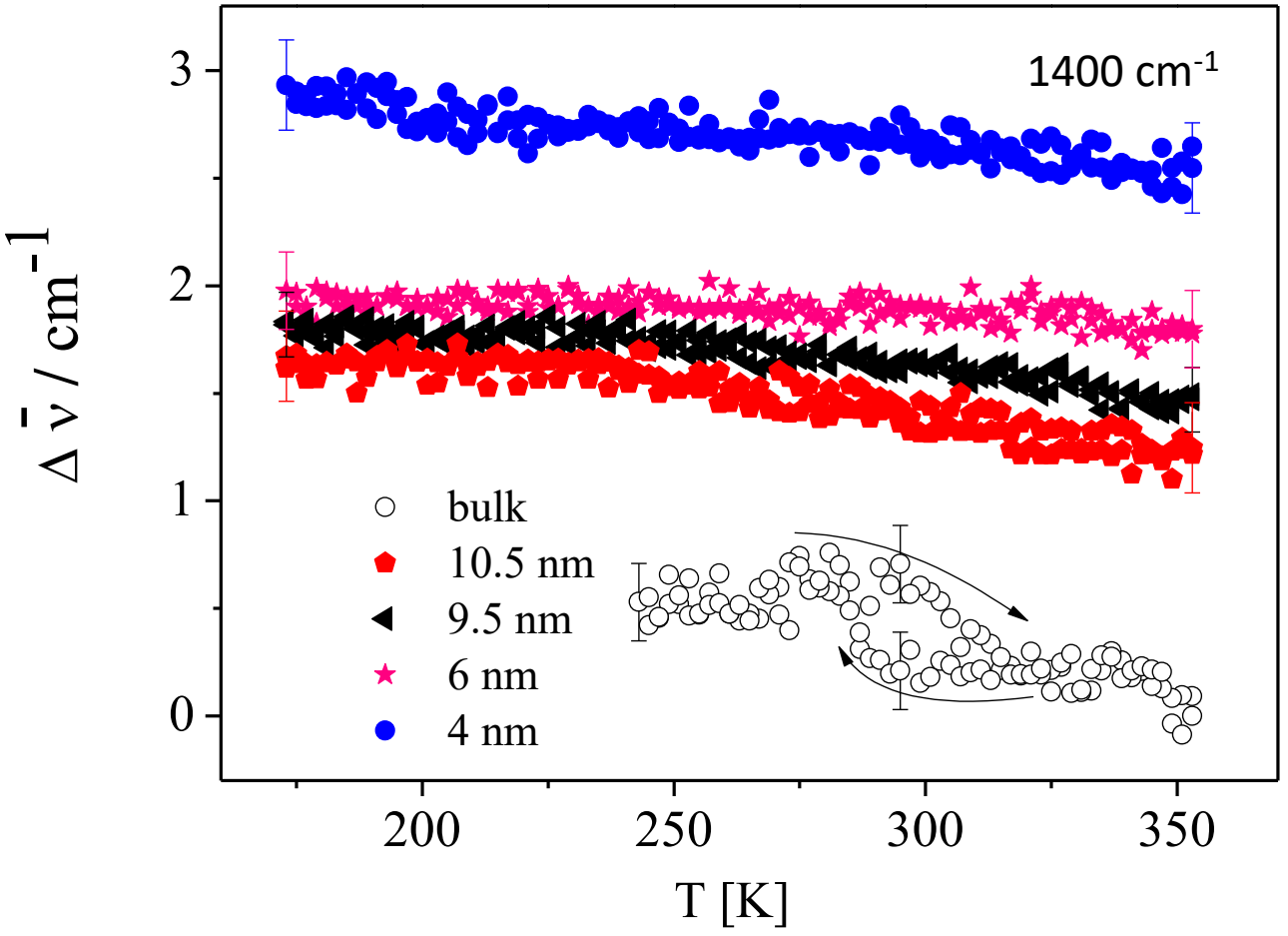
Vibrational dynamics of 7BT in bulk and in 6nm pores

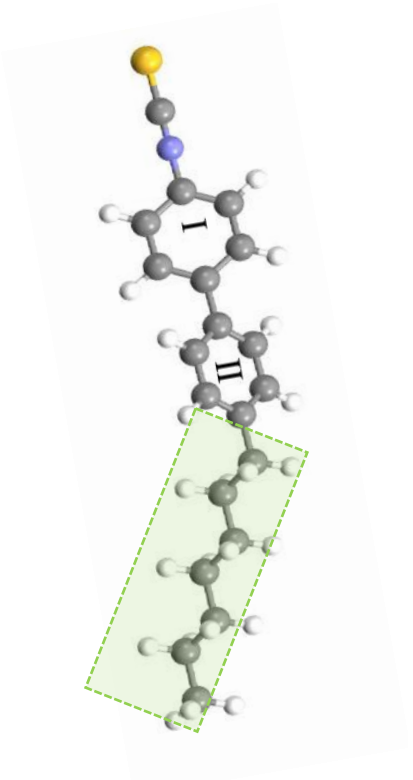




The SmE phase does not occur in nano-pores.

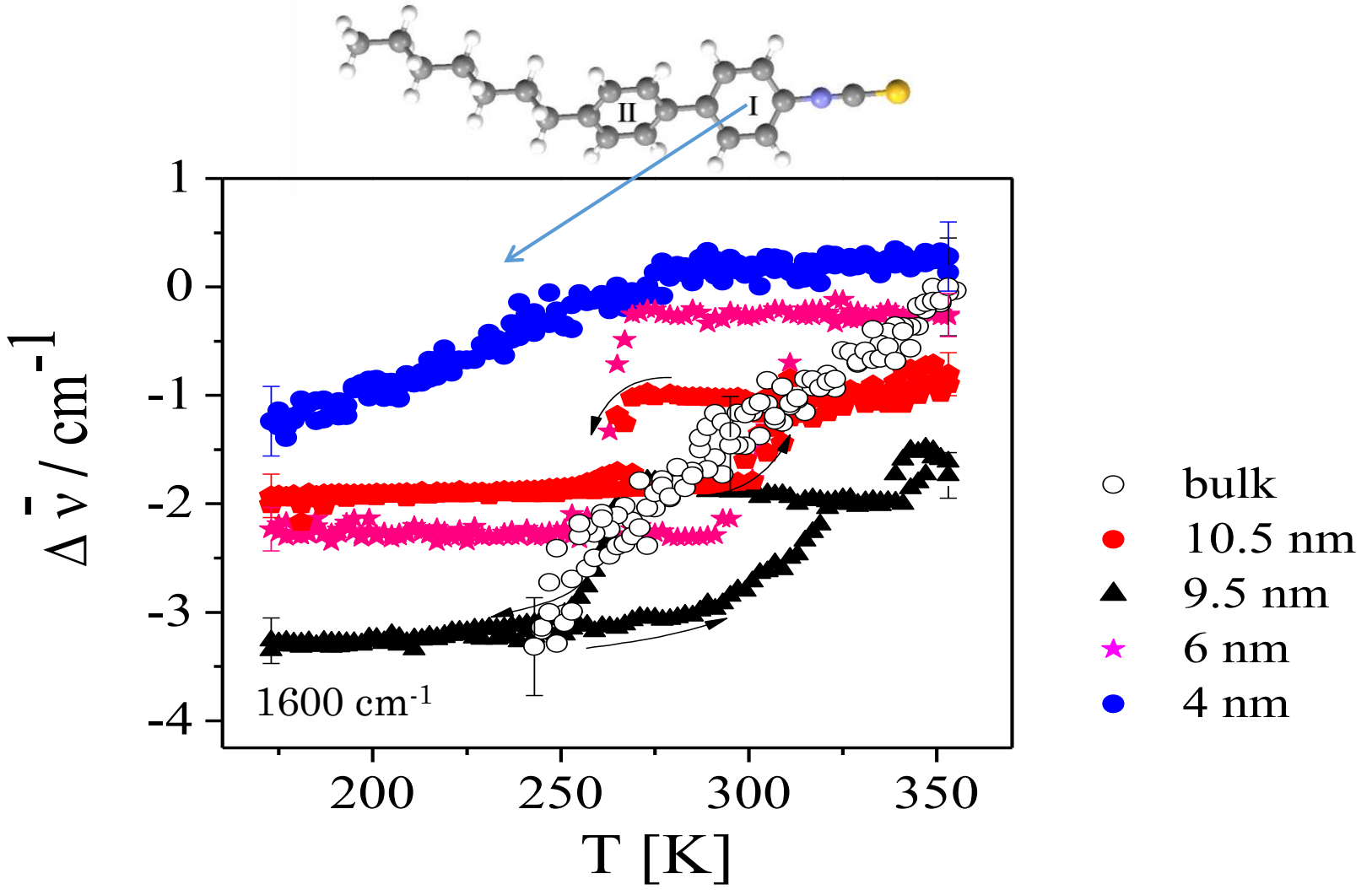
Shift of the absorption bands with respect to the spectral position in isotropic bulk





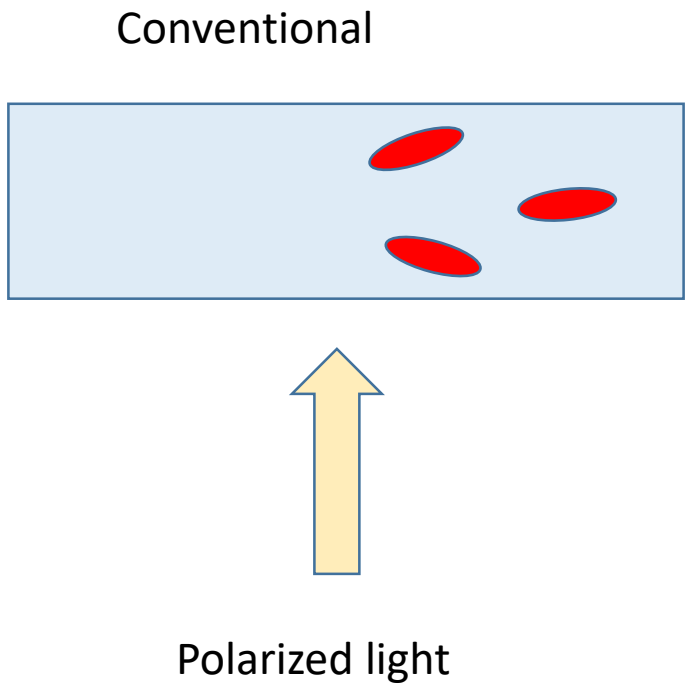
Because the crystallization process is fully suppressed in pores with a diameter of only a few nanometers, the positions of the bands related to vibration of CH2 groups do not show thermal hysteresis.

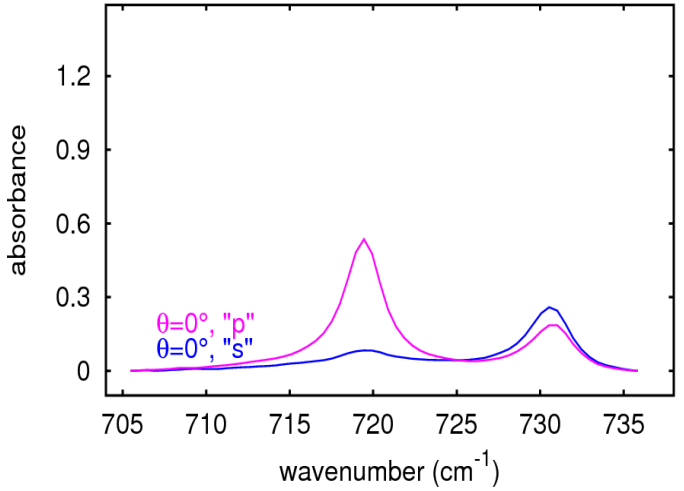
Shift of the absorption bands with respect to the spectral position in isotropic bulk

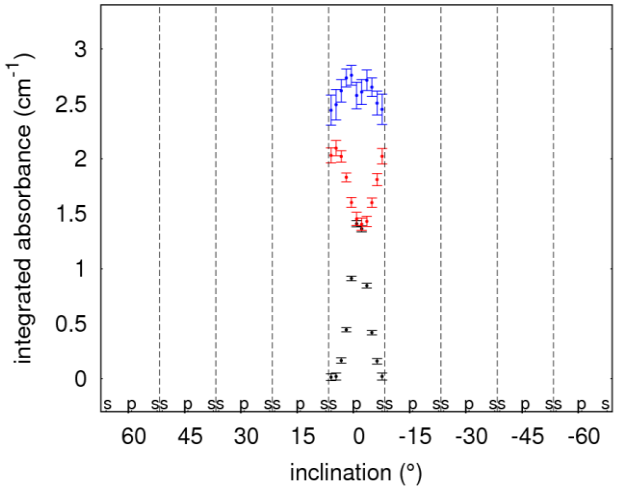


The molecular cores have tendency to order upon cooling.

Infrared transition moment orientational analysis

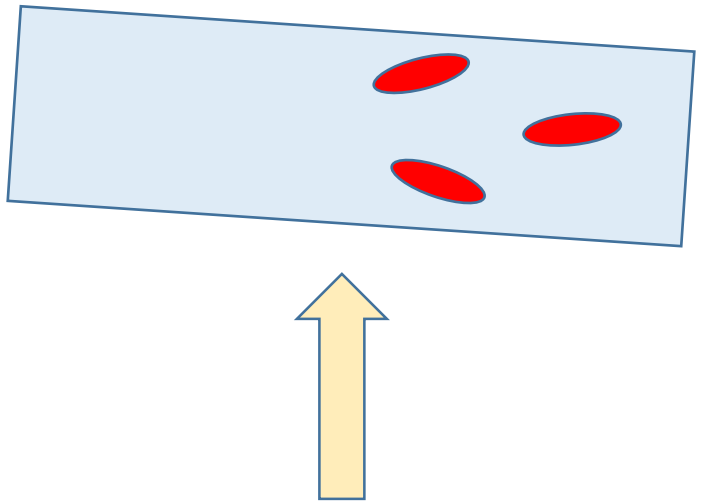




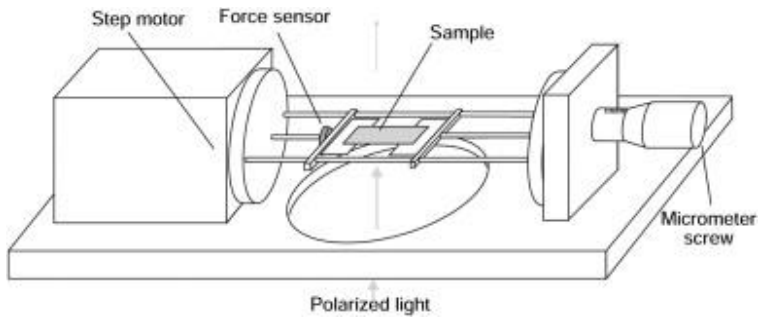


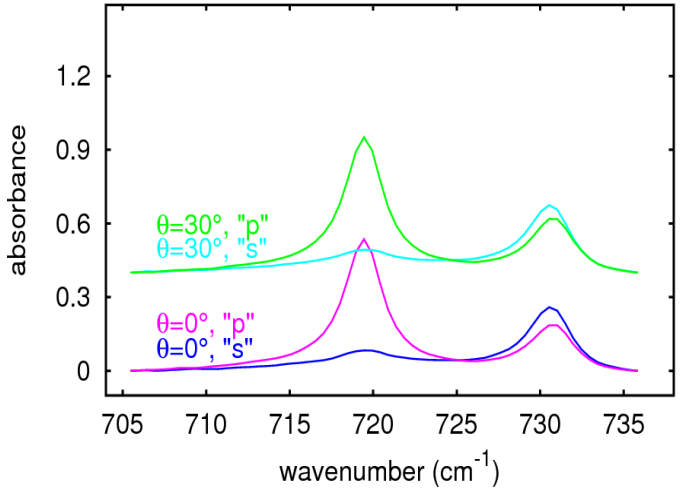
W. Kossack, P. Papadopoulos, P. Heinze, H. Finkelmann and F. Kremer. Macromolecules. 2010, Vol. 43, pp. 7532-7539.

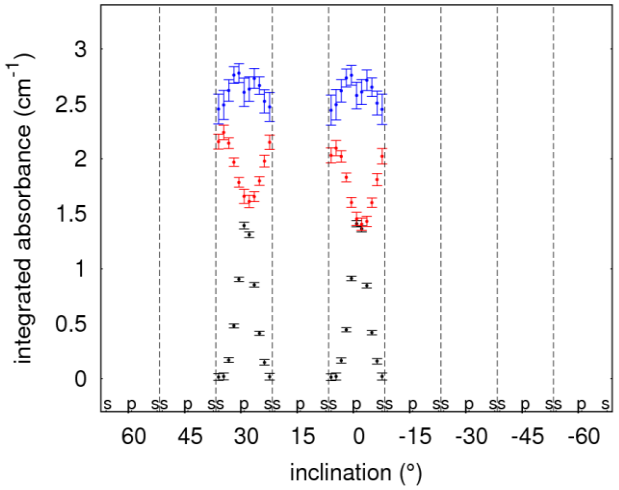
Infrared transition moment orientational analysis



Polarized light







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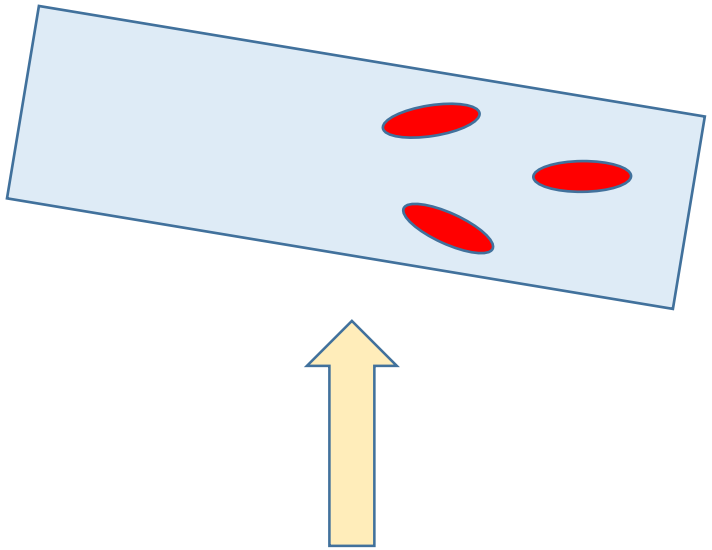
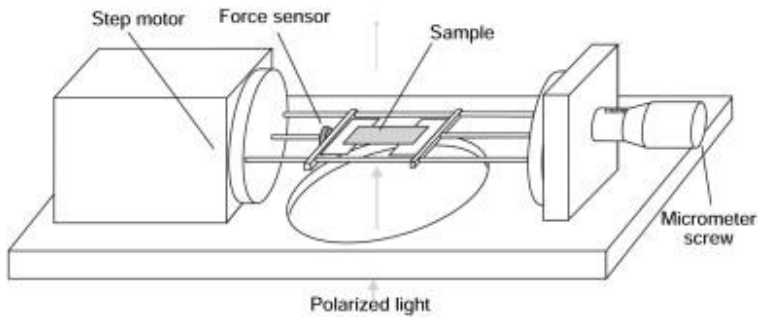
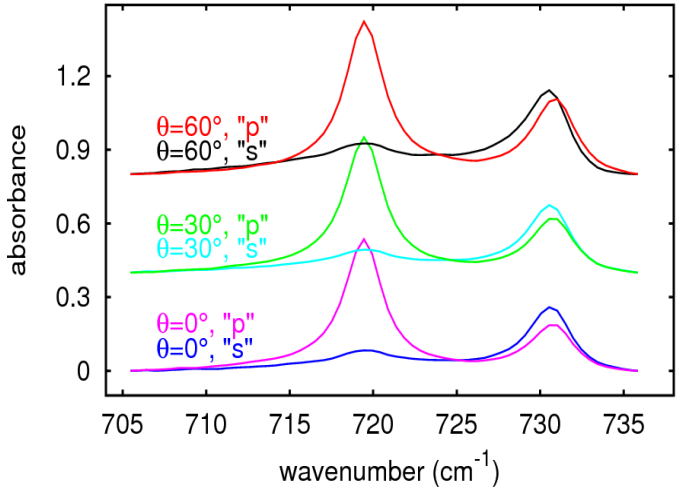
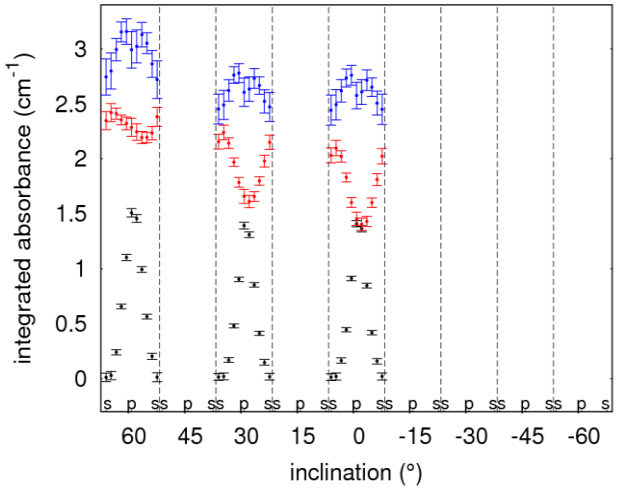
Infrared transition moment orientational analysis



Polarized light

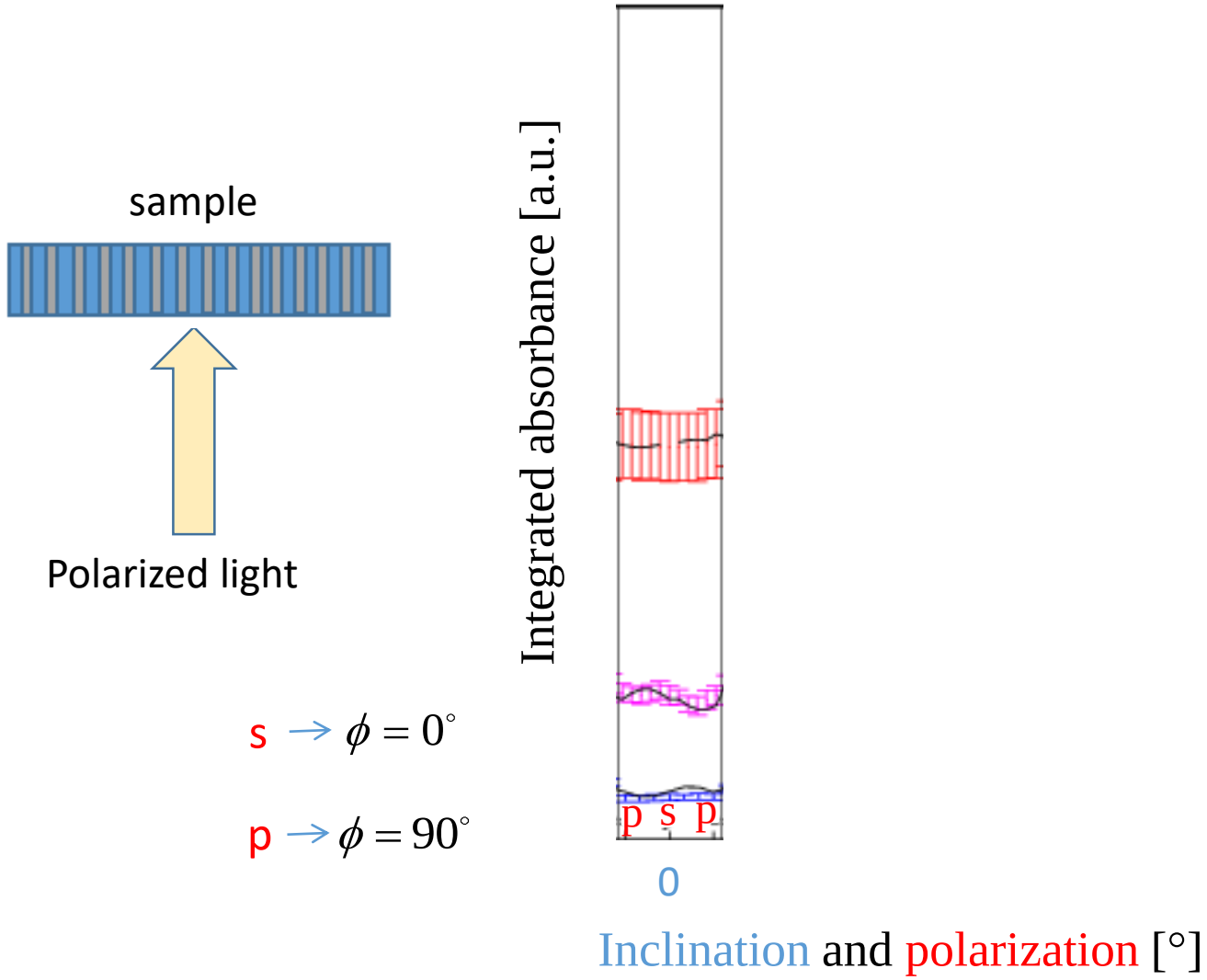






By measuring both, the polarisation and the inclination dependence of the IR spectra the three dimensional orientational distribution of the different molecular moieties is determined.

Infrared transition moment orientational analysis for 7BT LC in pores



M. Jasiurkowska, W. Kossack, R. Ene, C. Iacob, W. Kipnusu, P. Papadopoulos, J. R. Sangoro, M. Massalska-Arodź, F. Kremer, Soft Matter, 8, (2012) 5194

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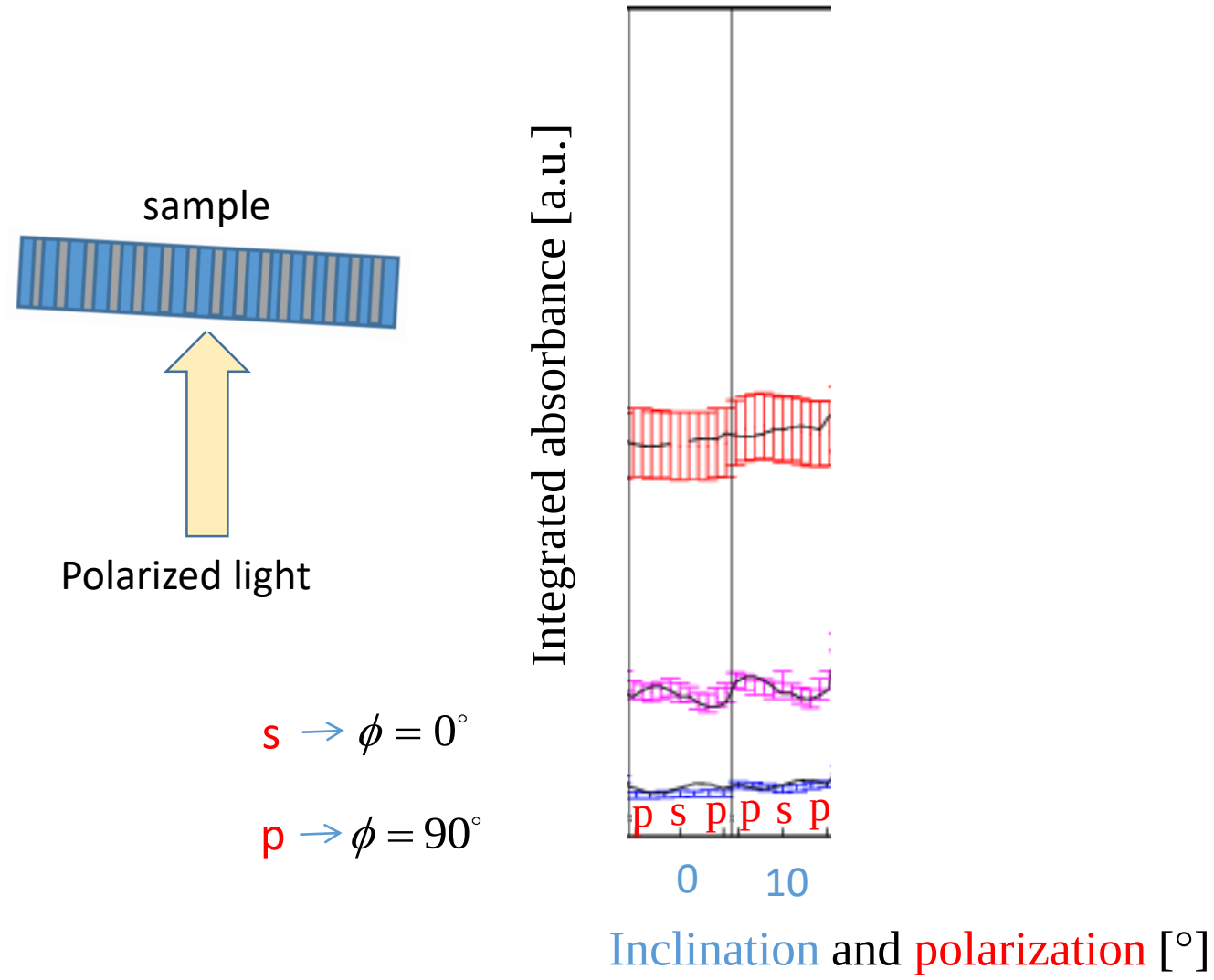

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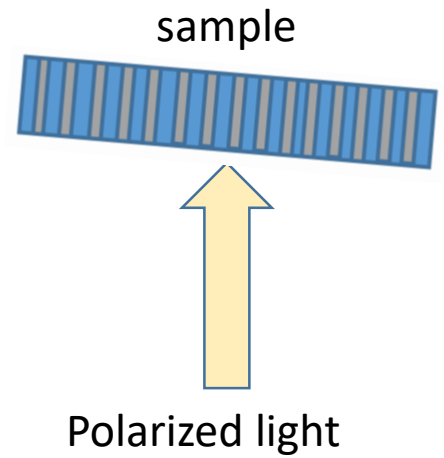
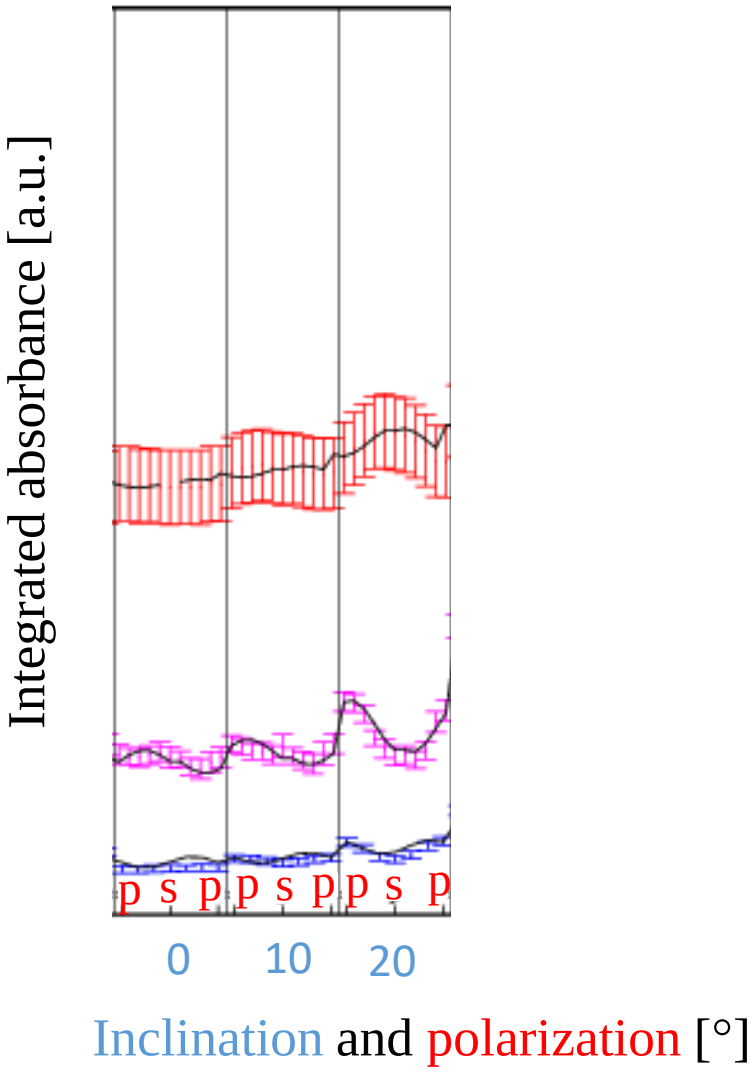


s $\rightarrow \phi = 0^{\circ}$

p $\rightarrow \phi = 90^{\circ}$



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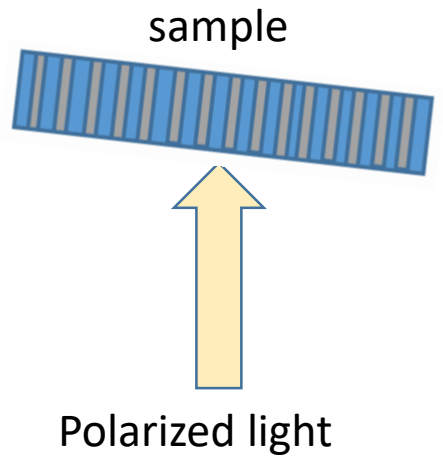

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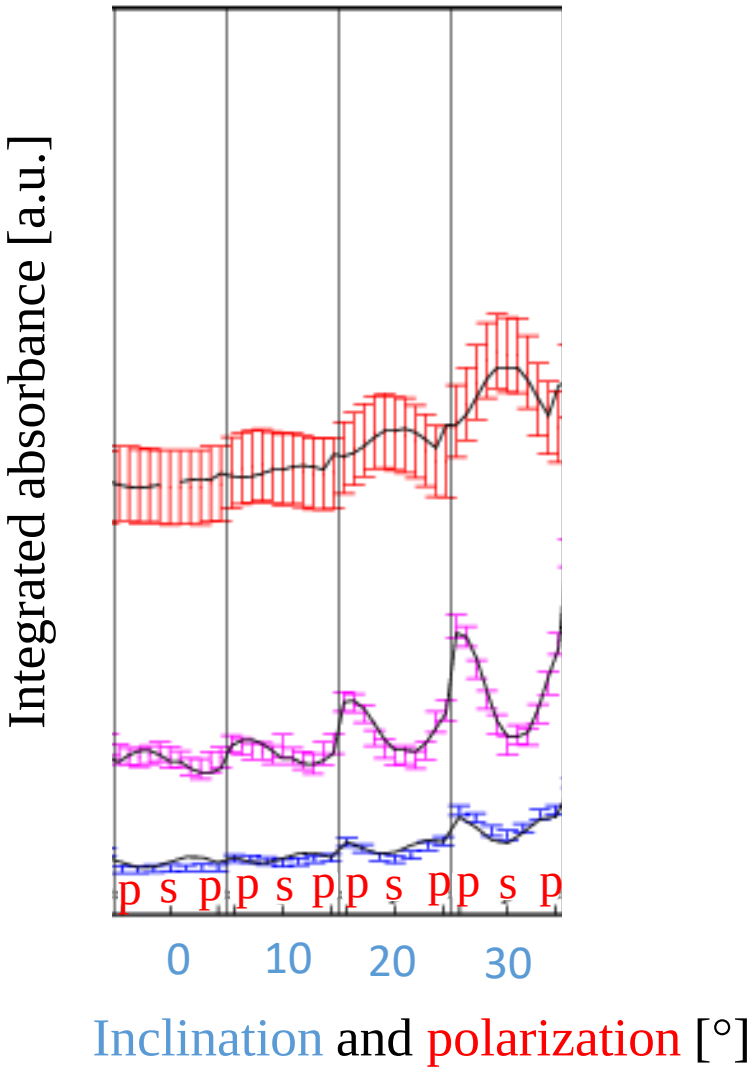

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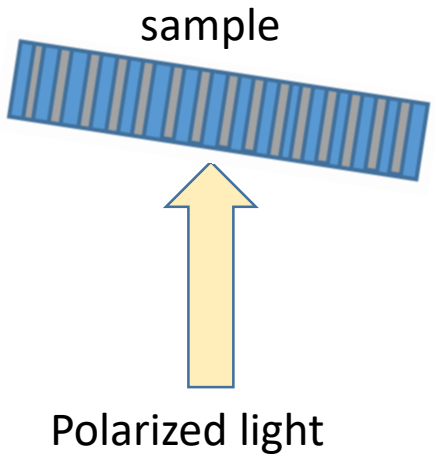

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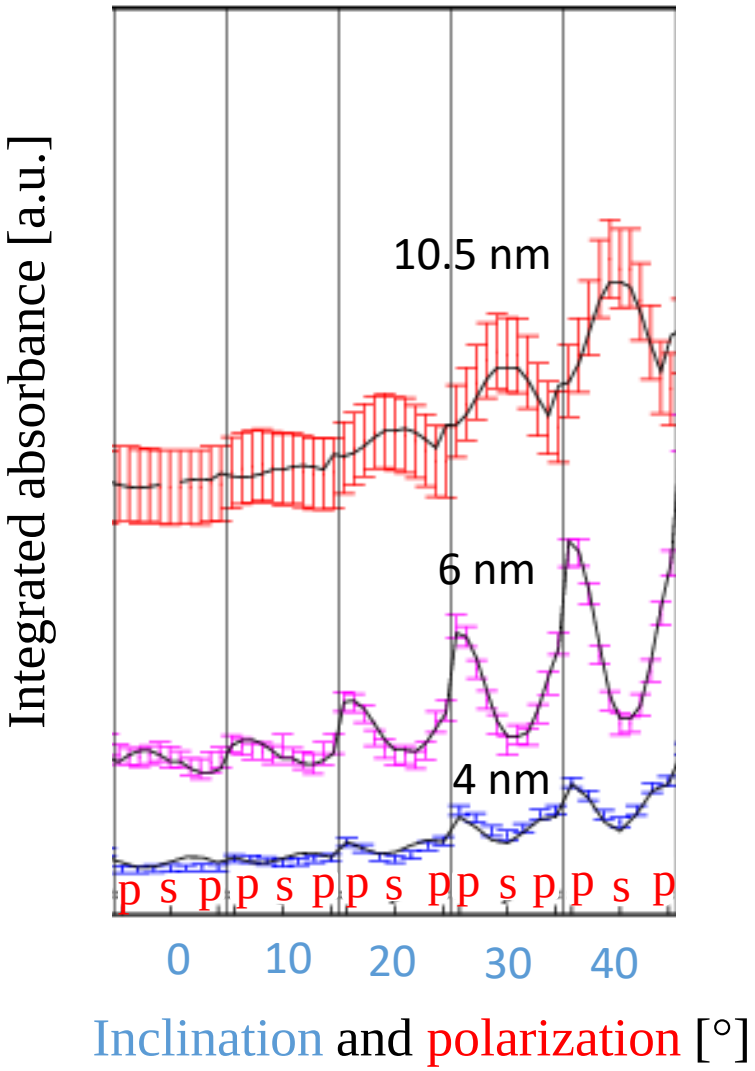
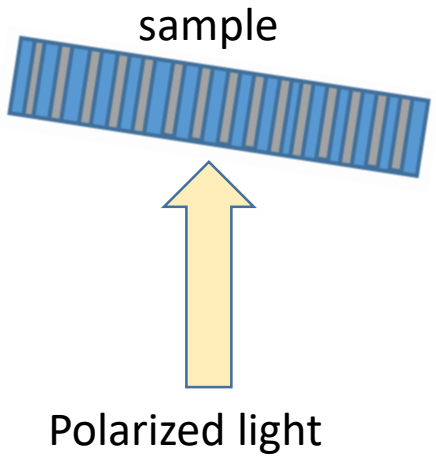


The average direction of orientation of molecular cores changed from a radial orientation (perpendicular to the pore walls) to an axial orientation (along the long axis of the pores) with decreasing pore diameter.

Infrared transition moment orientational analysis for 7BT LC in pores



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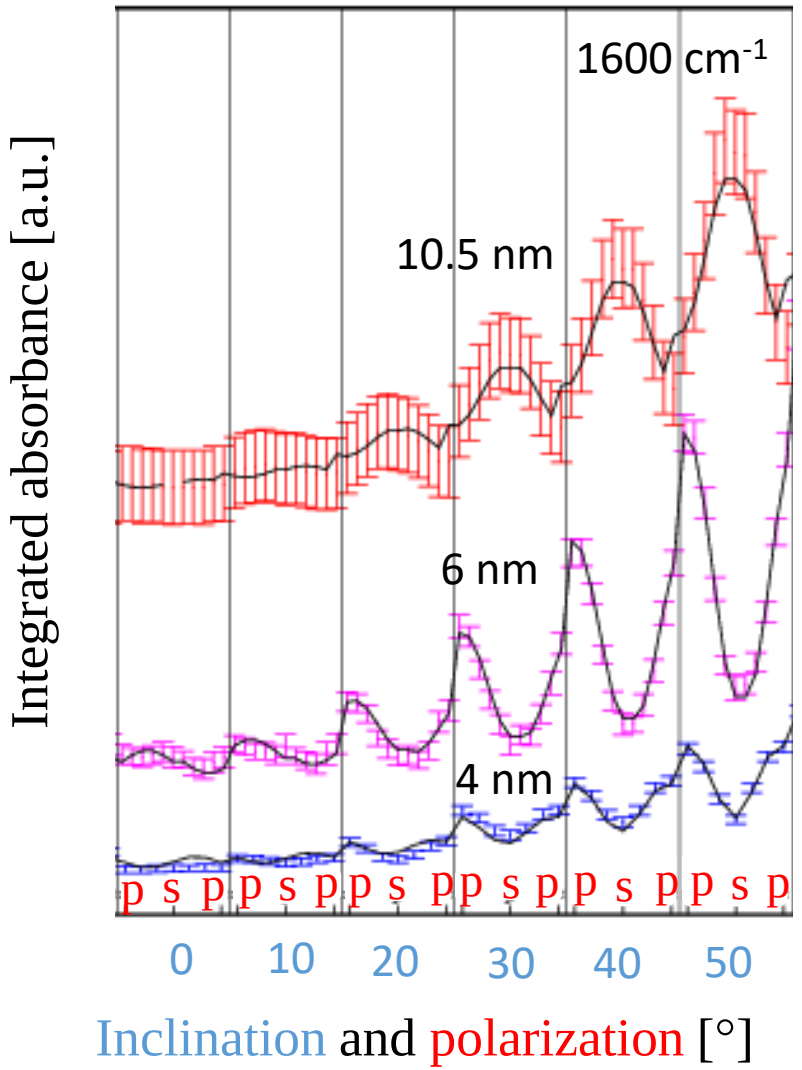

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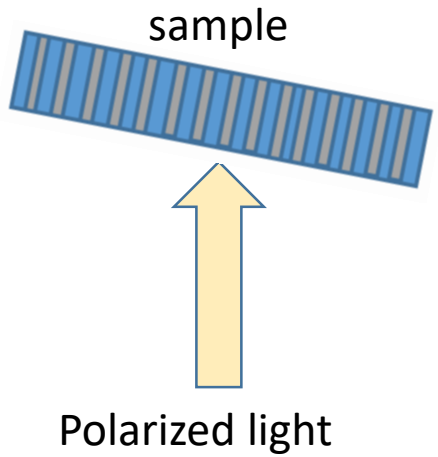

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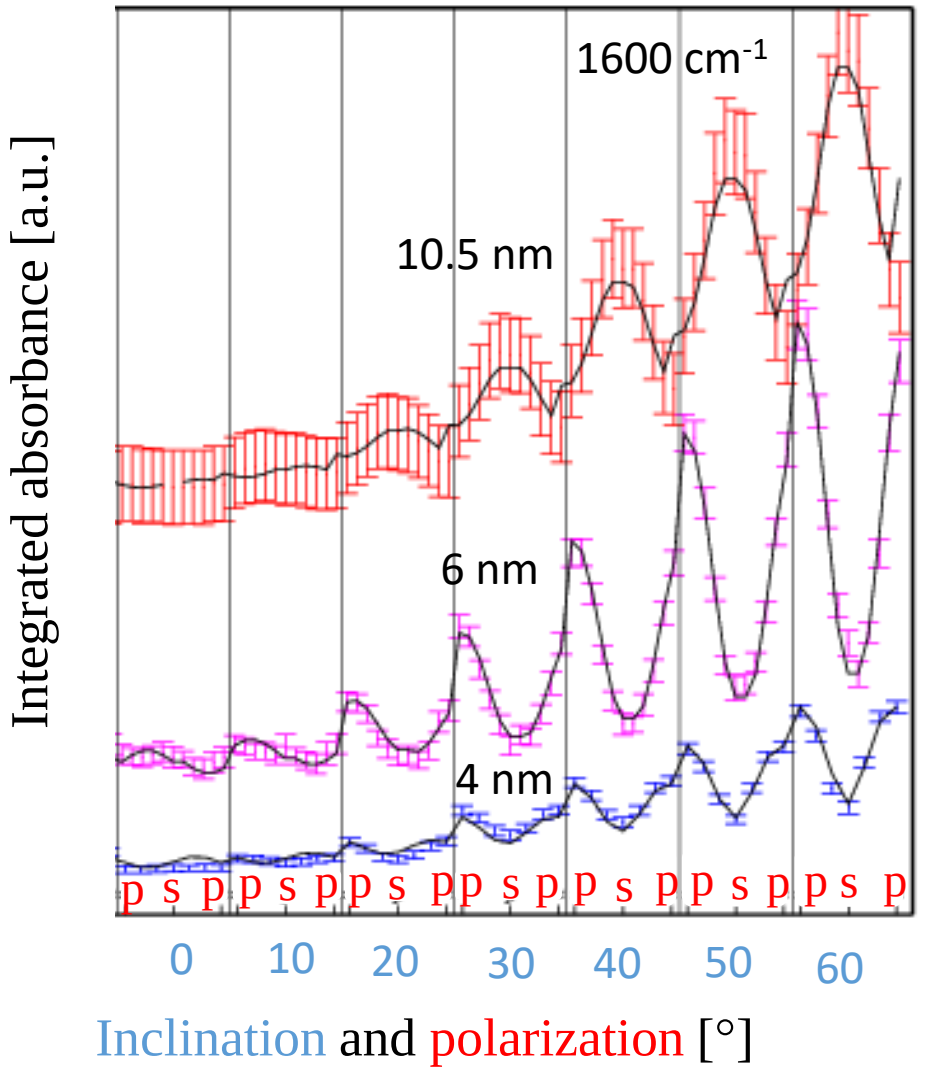

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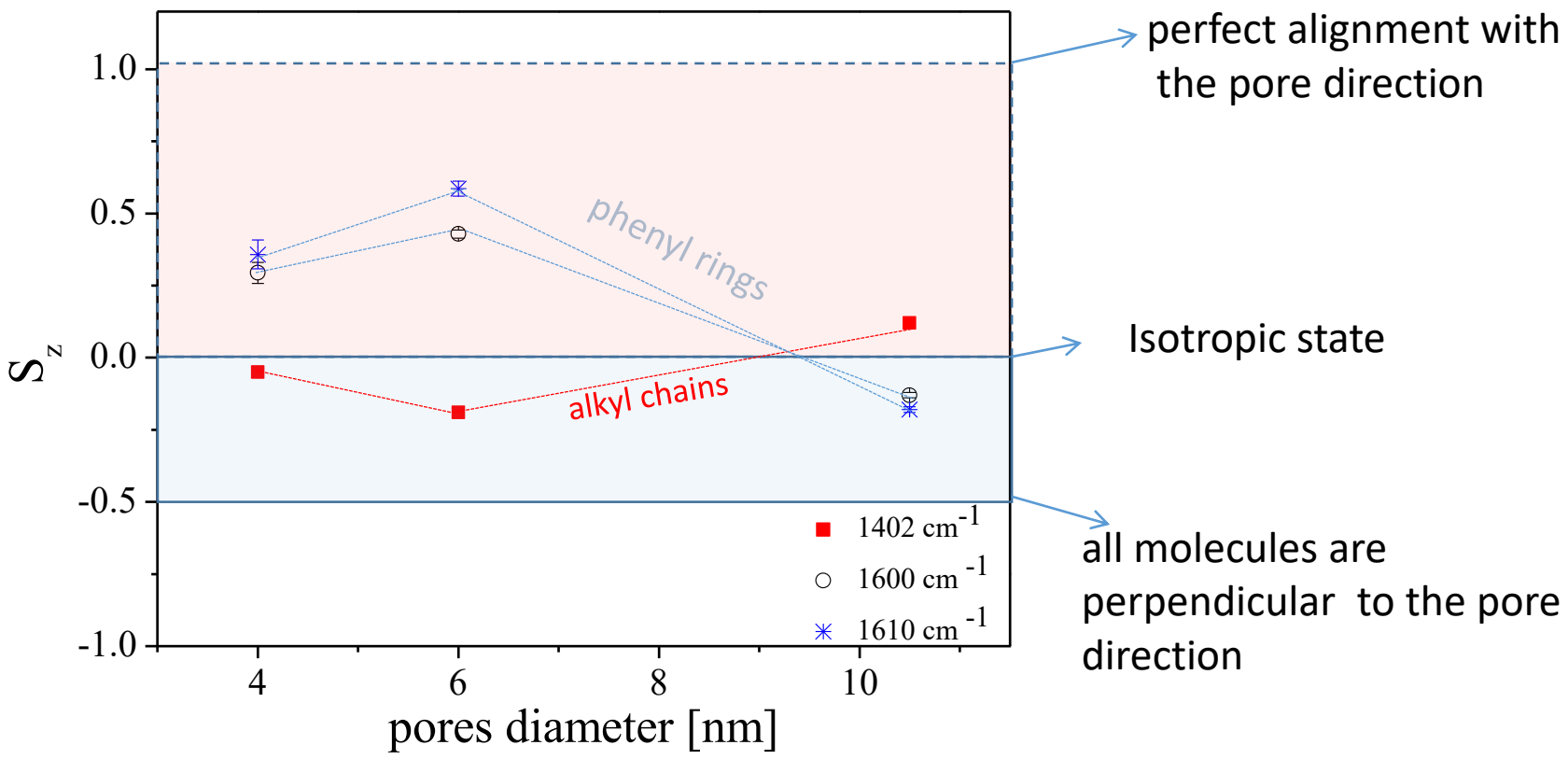
Order parameter measured with respect to the pore direction

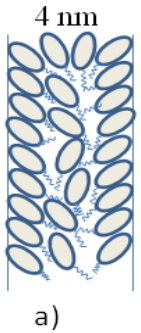


in 10.5 nm pores ➡ molecular cores ⊥ pore walls

in 4nm and 6 nm pores ➡ molecular cores ∥ pore walls

M. Jasiurkowska, W. Kossack, R. Ene, C. Iacob, W. Kipnusu, P. Papadopoulos, J. R. Sangoro, M. Massalska-Arodź, F. Kremer, Soft Matter, 8, (2012) 5194

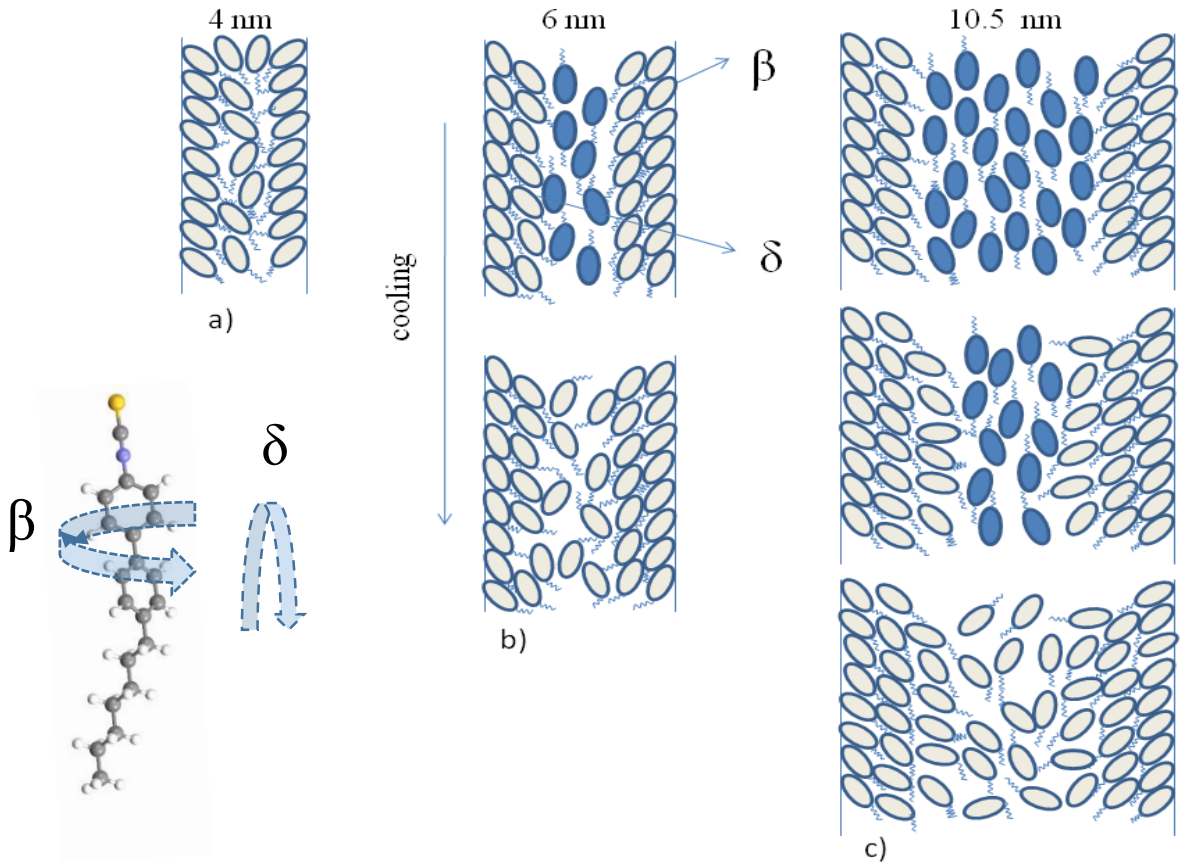
Molecular arrangement of 7BT LC in nanopores



a) The molecules are partially immobilized and only librational fluctuations take place.

***M. Jasiurkowska**, W. Kossack, R. Ene, C. Iacob, W. Kipnusu, P. Papadopoulos, J. R. Sangoro, M. Massalska-Arodź, F. Kremer, Soft Matter, 8, (2012) 5194*

Molecular arrangement of 7BT LC in nanopores



a) The molecules are partially immobilized and only librational fluctuations take place. b) and c) Upon cooling, a layer of ordered L.C. molecules is formed at the walls.

M. Jasiurkowska, W. Kossack, R. Ene, C. Iacob, W. Kipnusu, P. Papadopoulos, J. R. Sangoro, M. Massalska-Arodź, F. Kremer, Soft Matter, 8, (2012) 5194

How different forms of confinement affect the molecular dynamics and crystallization process of LCs ?

„*Hard*" confinement
LCs in nanoporous matrix
(SiO_2, AAO)

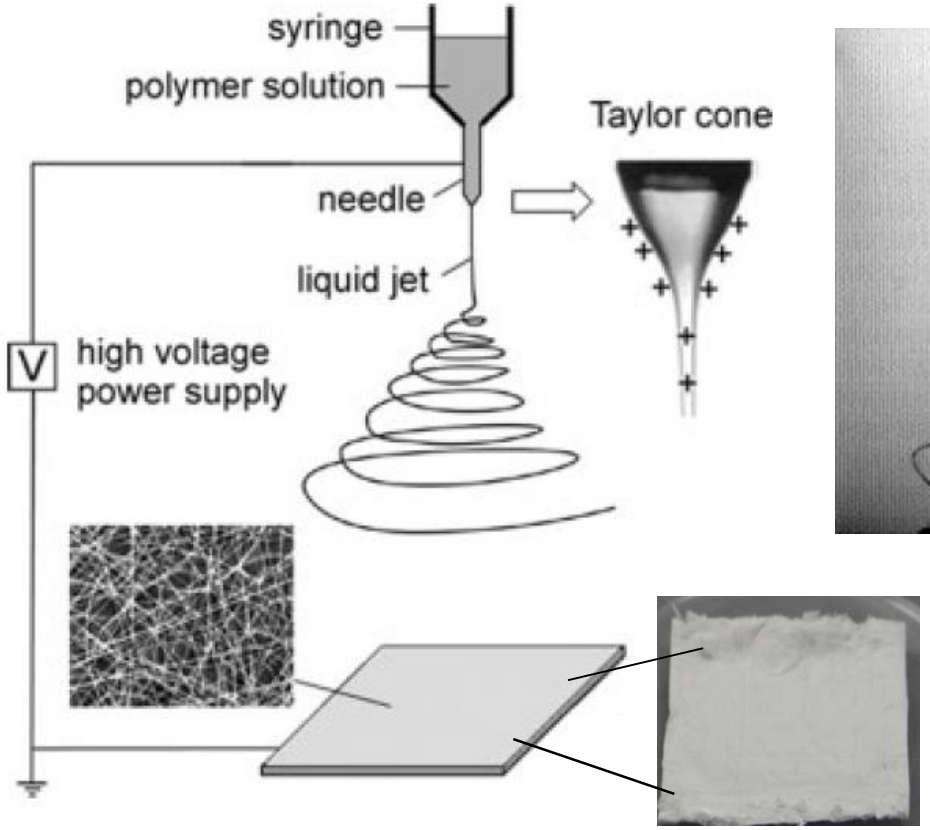
VS.

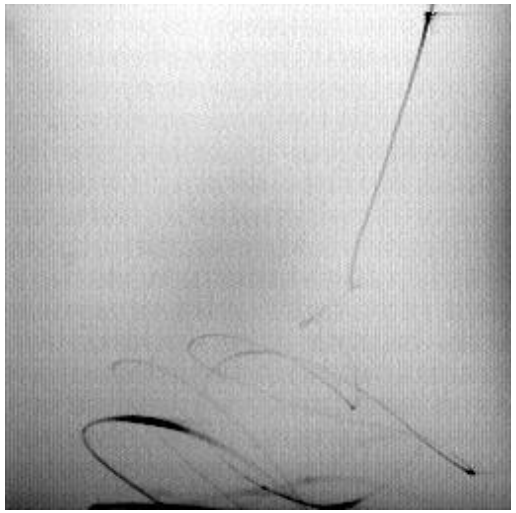
„Soft" confinement
LCs in polymer matrix

- Acceleration of motions of the *volume* molecules with respect to the bulk
- New relaxation processes
- Suppression of the crystallization with decreasing pores size

?

Electrospun fibers

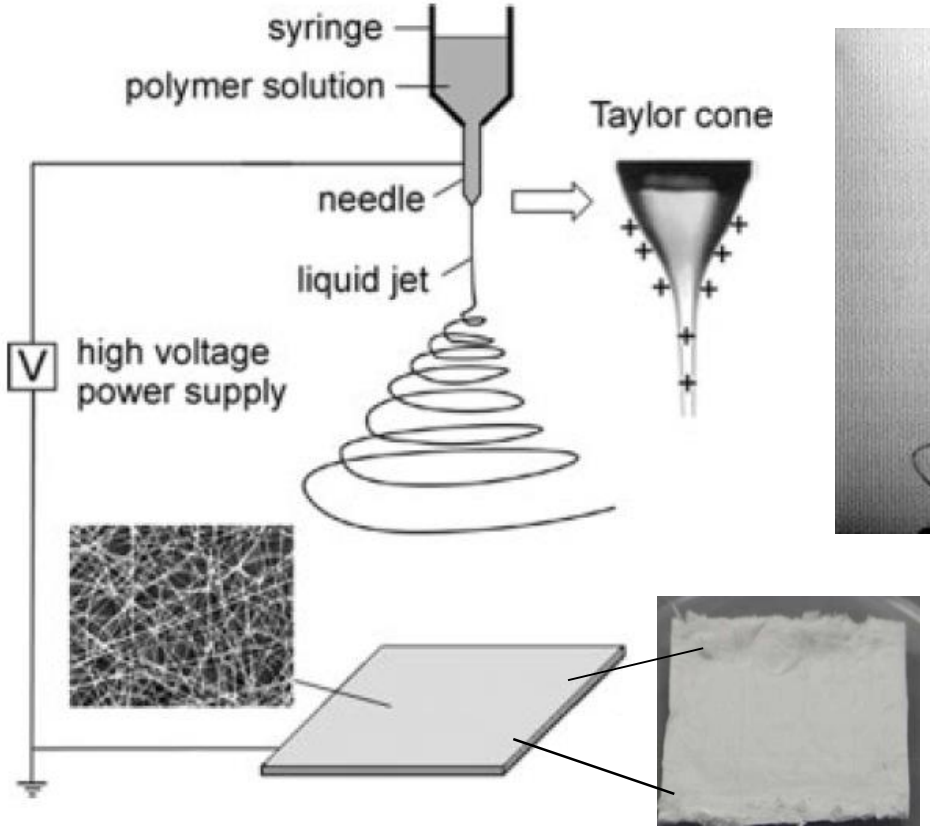


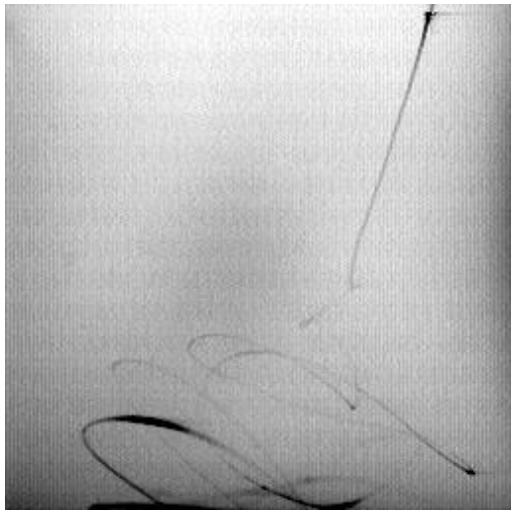


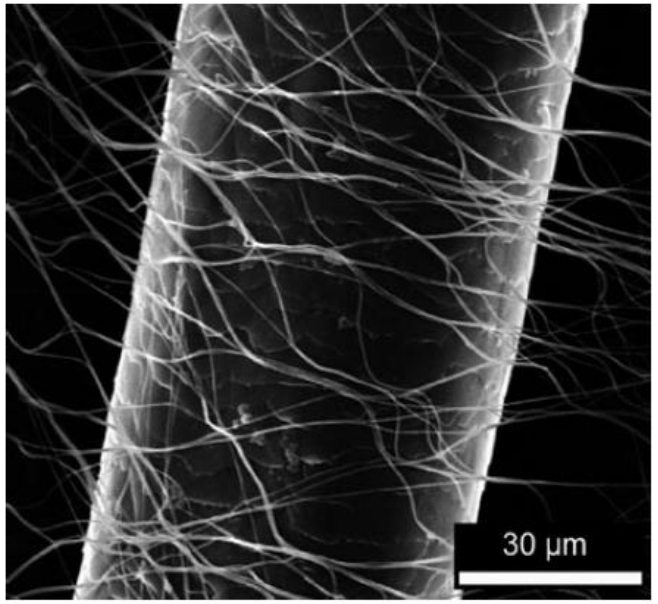
Electrospining setup at IFJ

Electrospinning is a high-voltage driven technique used to produce long, continuous fibres with diameter ranging from few nanometers to several micrometers

Electrospun fibers







Scanning electron microscope (SEM) image of a human hair surrounded by electrospun fibers of poly(vinyl alcohol) (PVA).

Electrospinning is a high-voltage driven technique used to produce long, continuous fibres with diameter ranging from few nanometers to several micrometers

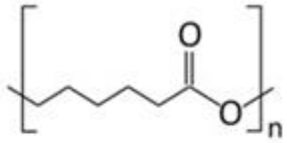
Why LCs/polymer composite fibers are interesting?

- 2008 - the first report on LC/polimer electrospun composite fibres by Lagerwall (Chem.Commun.,2008, 5420)

- LCs/polymer fibers show high potential for sensing applications (*Adv. Mater.* **2019**, 1902168)

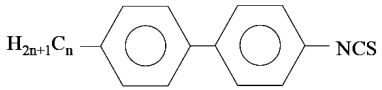
Preparation of PCL/6BT composite fibers

PCL - ε-polycaprolactone
Mw ~ 80 000 Da
10%wt - chloroform and ethanol (3:1)



+

6BT



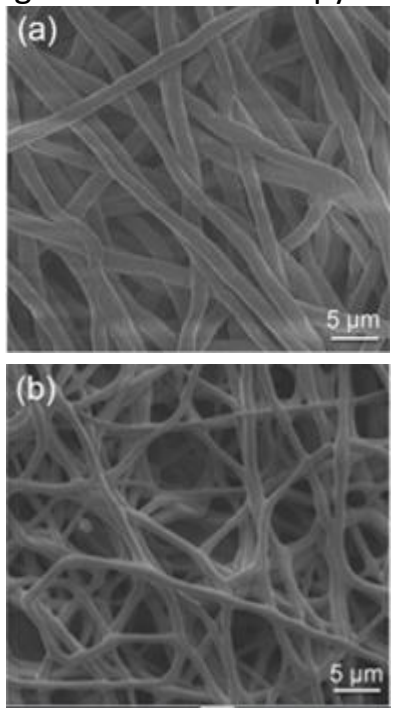
5, 10, 20, 30 mg/mol

ELECTROSPINNING

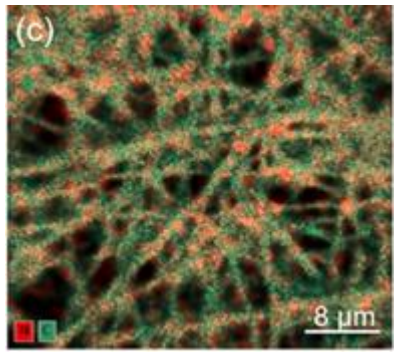
PCL/6BT: 4.7wt%, 8.2wt%, 17wt%
25.6 wt % composite fibers

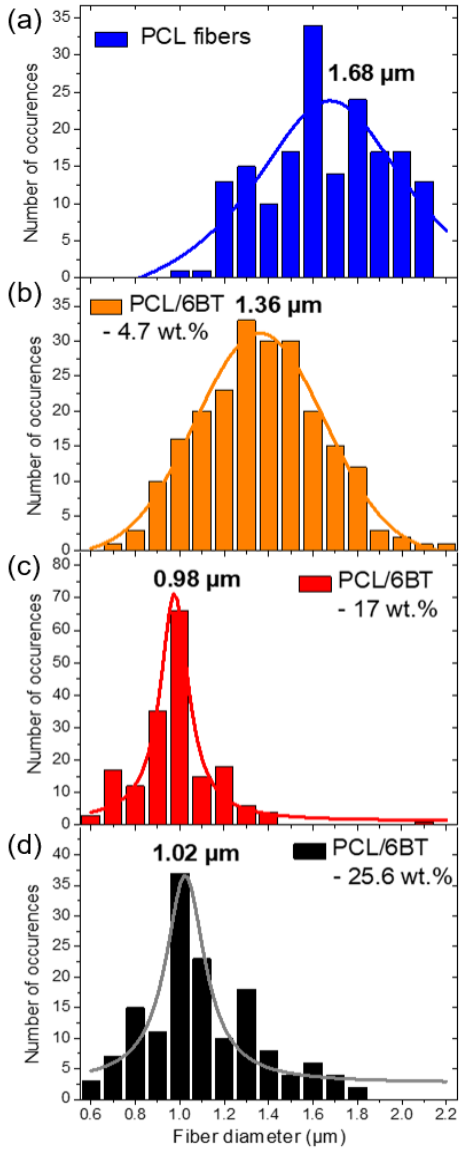
PCL mat

Scanning Electron Microscopy images



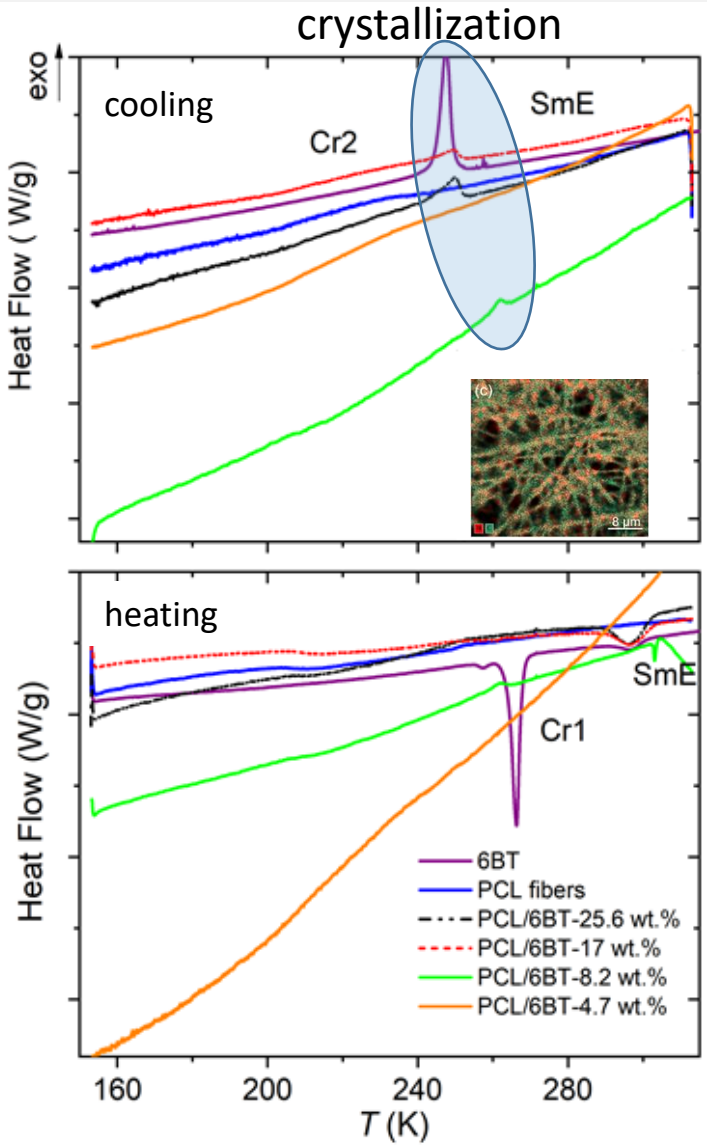
SEM-EDS map

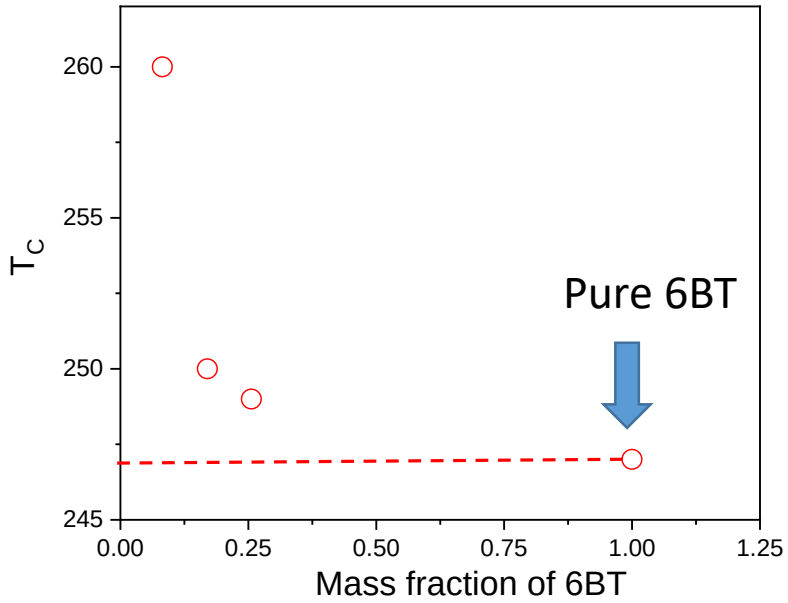




M. Jasiurkowska-Delaporte, E. Juszyńska-Gałązka, W. Sas, P. M. Zieliński, A. Baranowska-Korczyc, J. Mol. Liq., 331 (2021) 115817

Crystallization process of 6BT LC in constrained in fibers and pores





„Soft" confinement enhances the crystallization proces upon cooling.

M. Jasiurkowska-Delaporte, E. Juszyńska-Gałązka, W. Sas, P. M. Zieliński, A. Baranowska-Korczyc, J. Mol. Liq., 331 (2021) 115817

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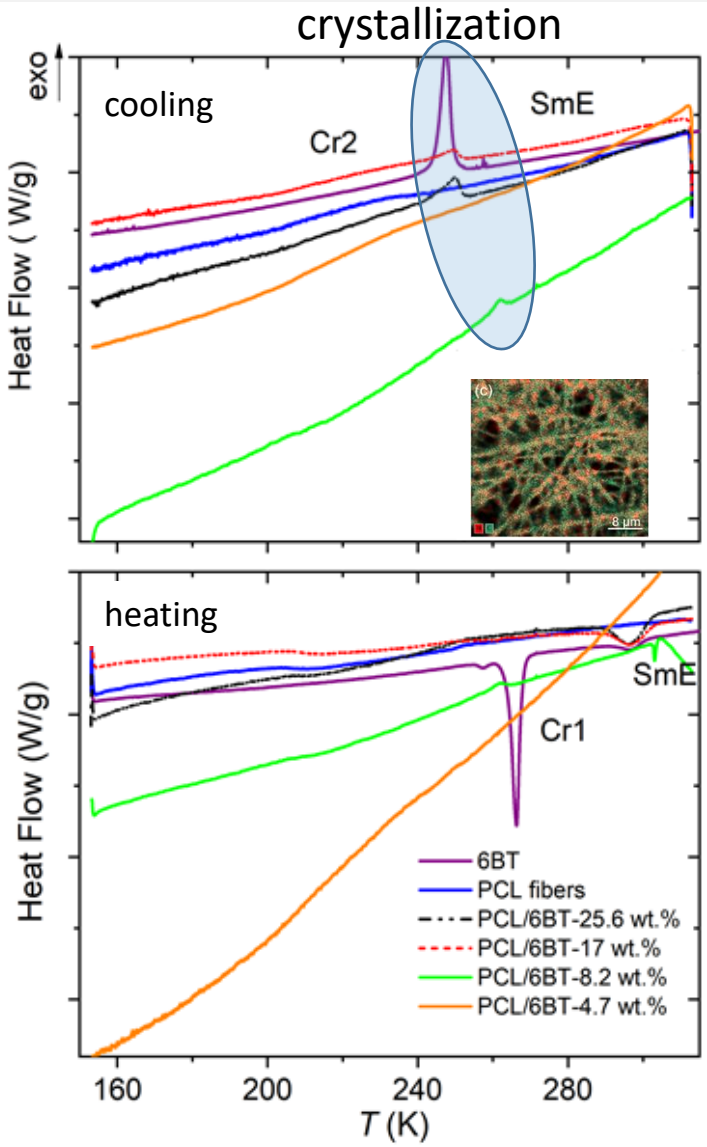
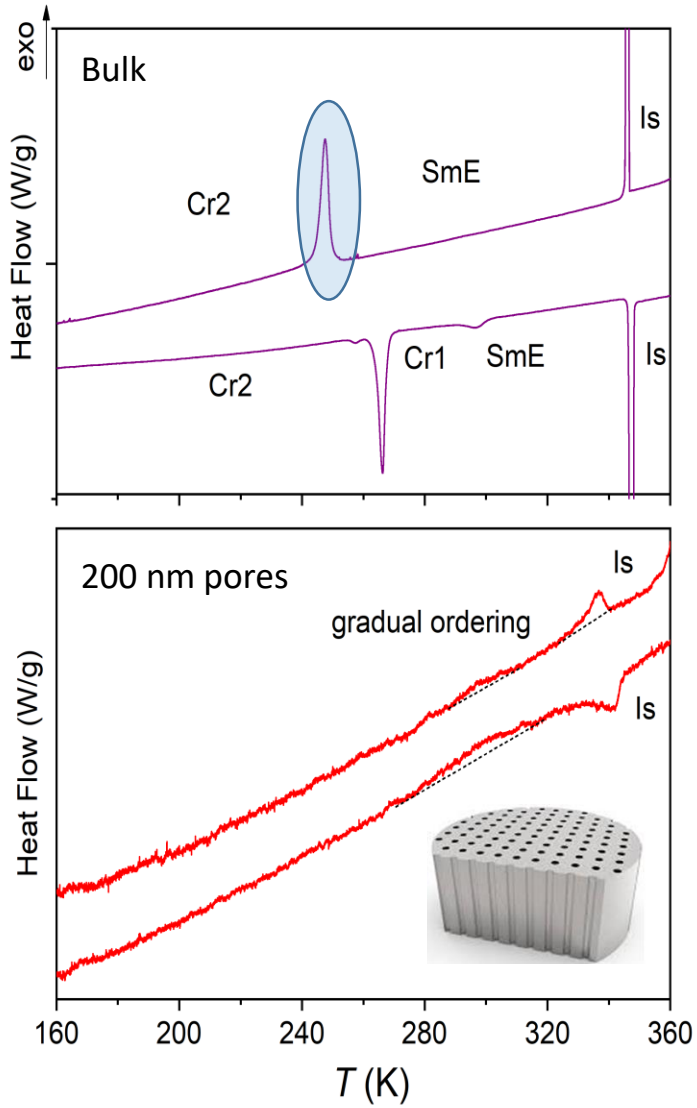
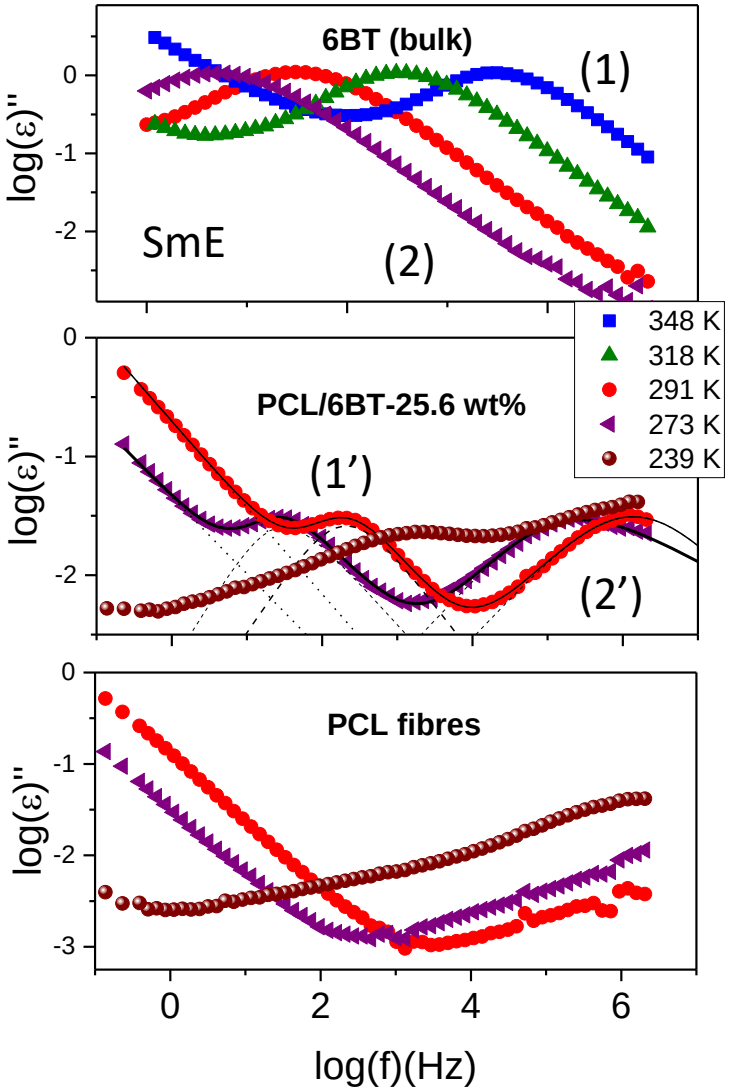
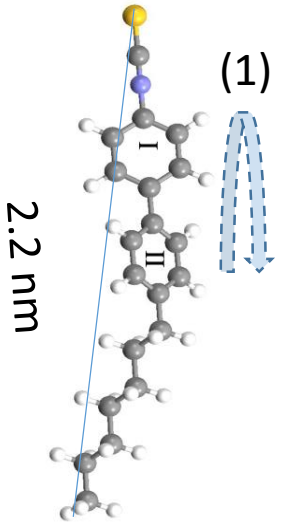




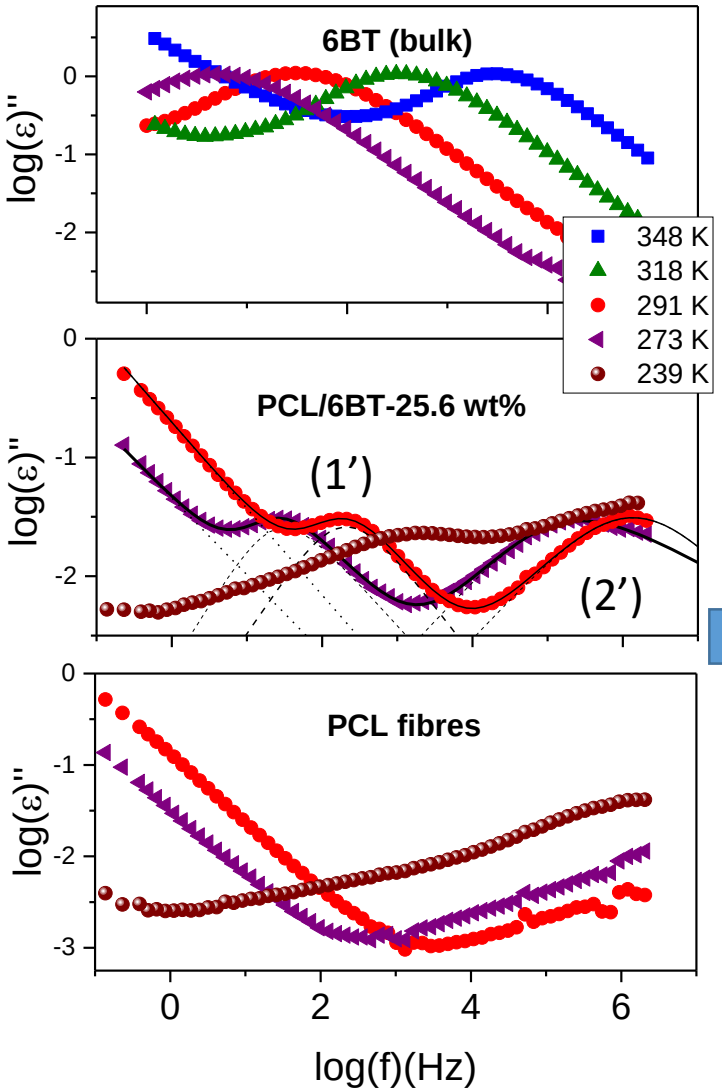
The crystallization process observed during the cooling of bulk 6BT is completely suppressed in pores.

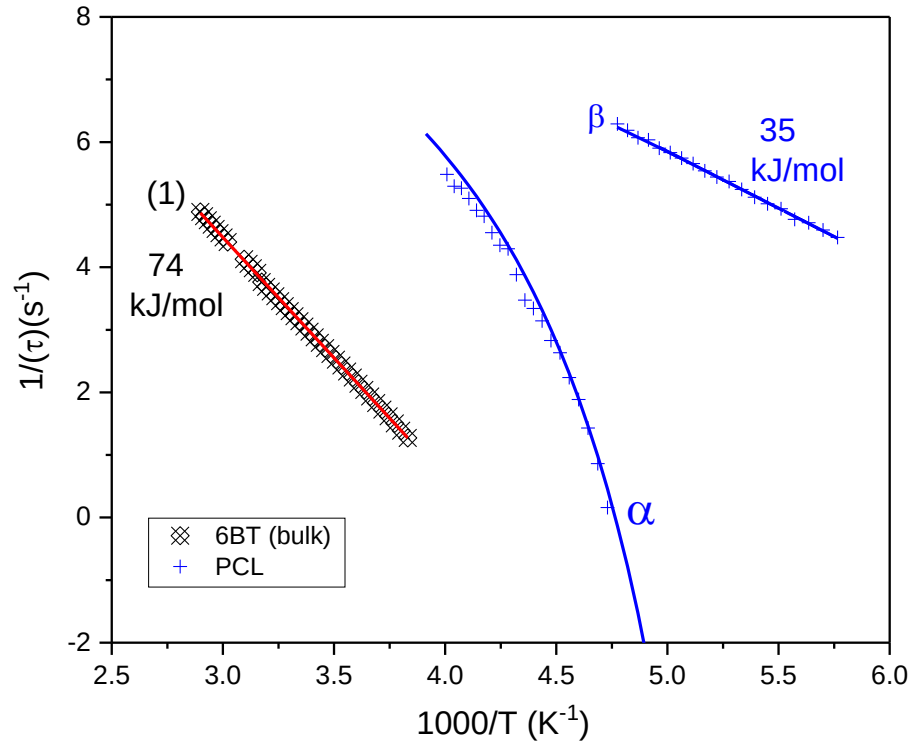
Molecular dynamics of 6BT liquid crystal under „soft” confinement





Molecular dynamics of 6BT liquid crystal under „soft” confinement





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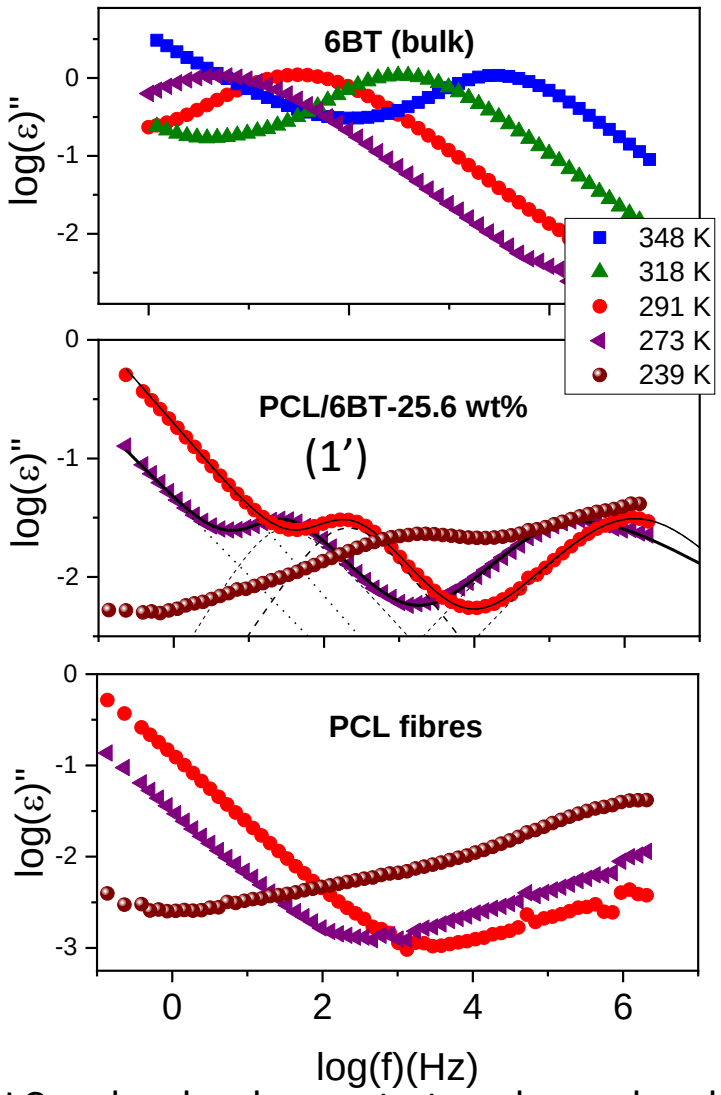
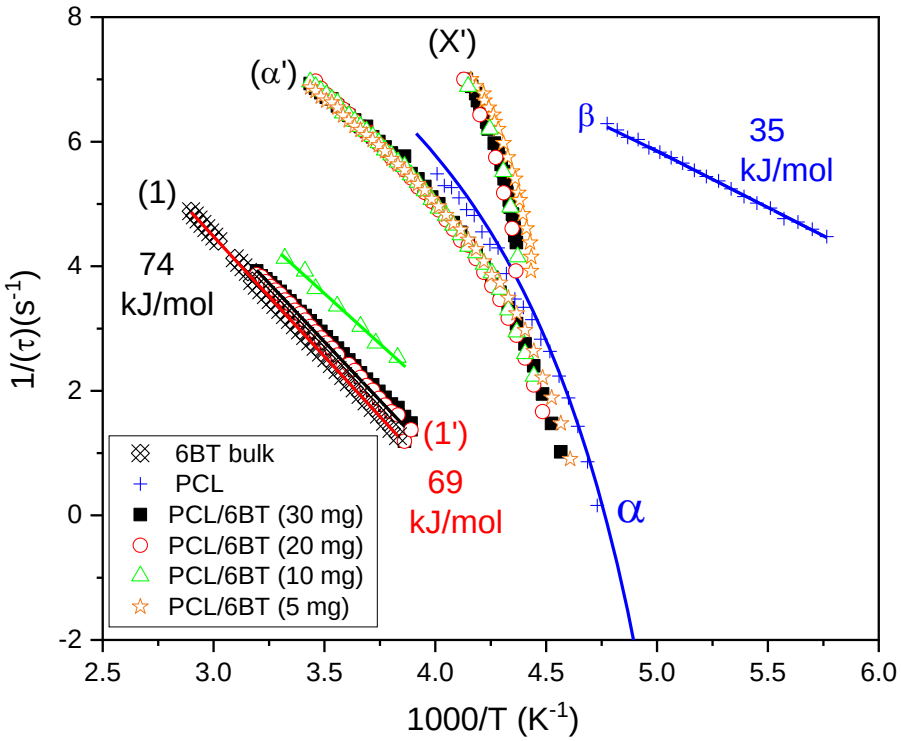
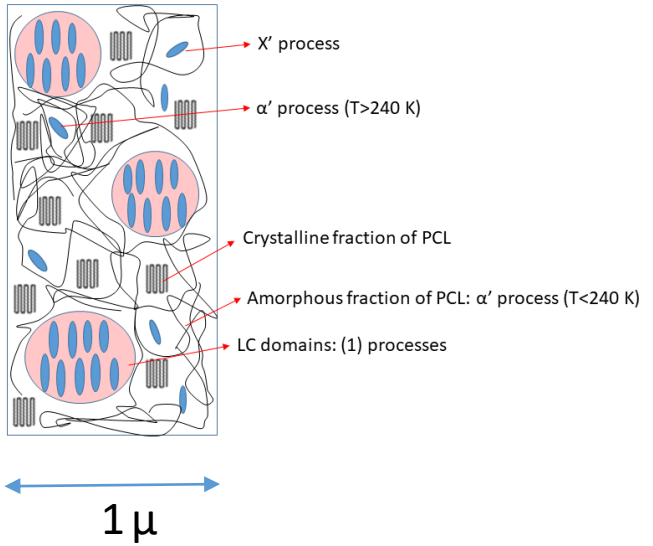




Composite fiber



LC molecules demonstrate enhanced mobility around their short axis while an additional relaxation process (α’) shows Arrhenius-like behaviour above 240 K then a sudden slowing of the (α’) process is found.

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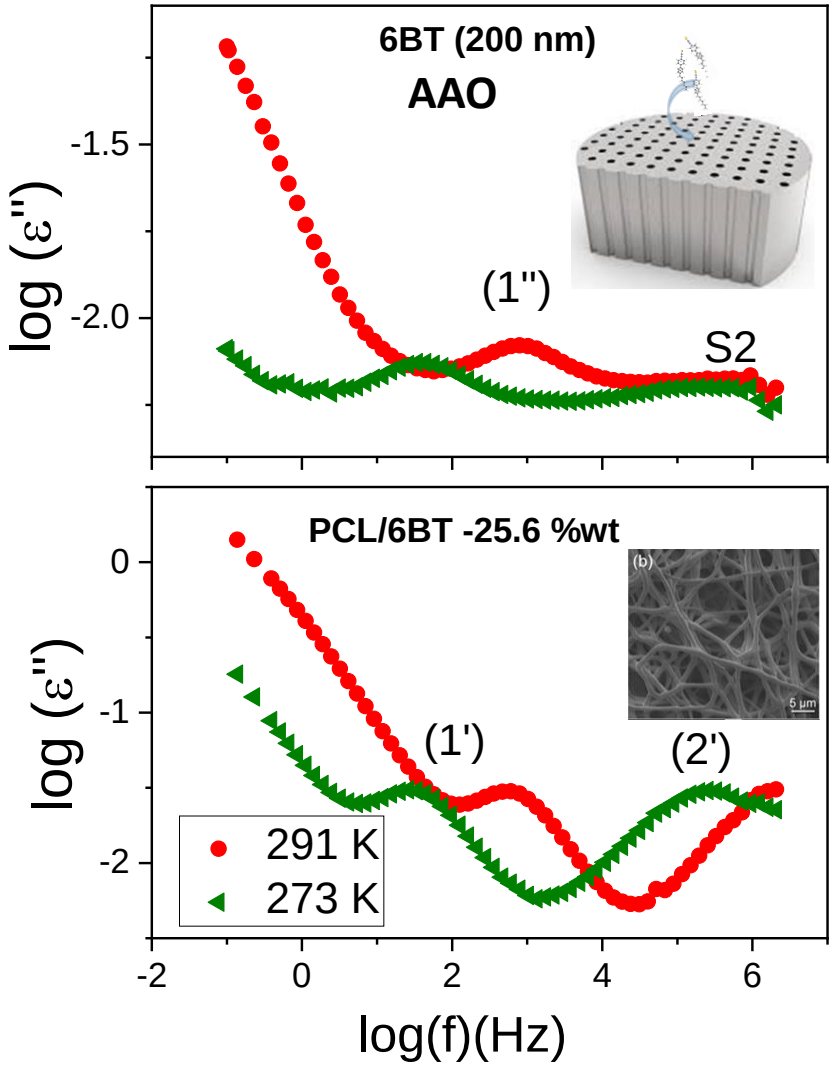
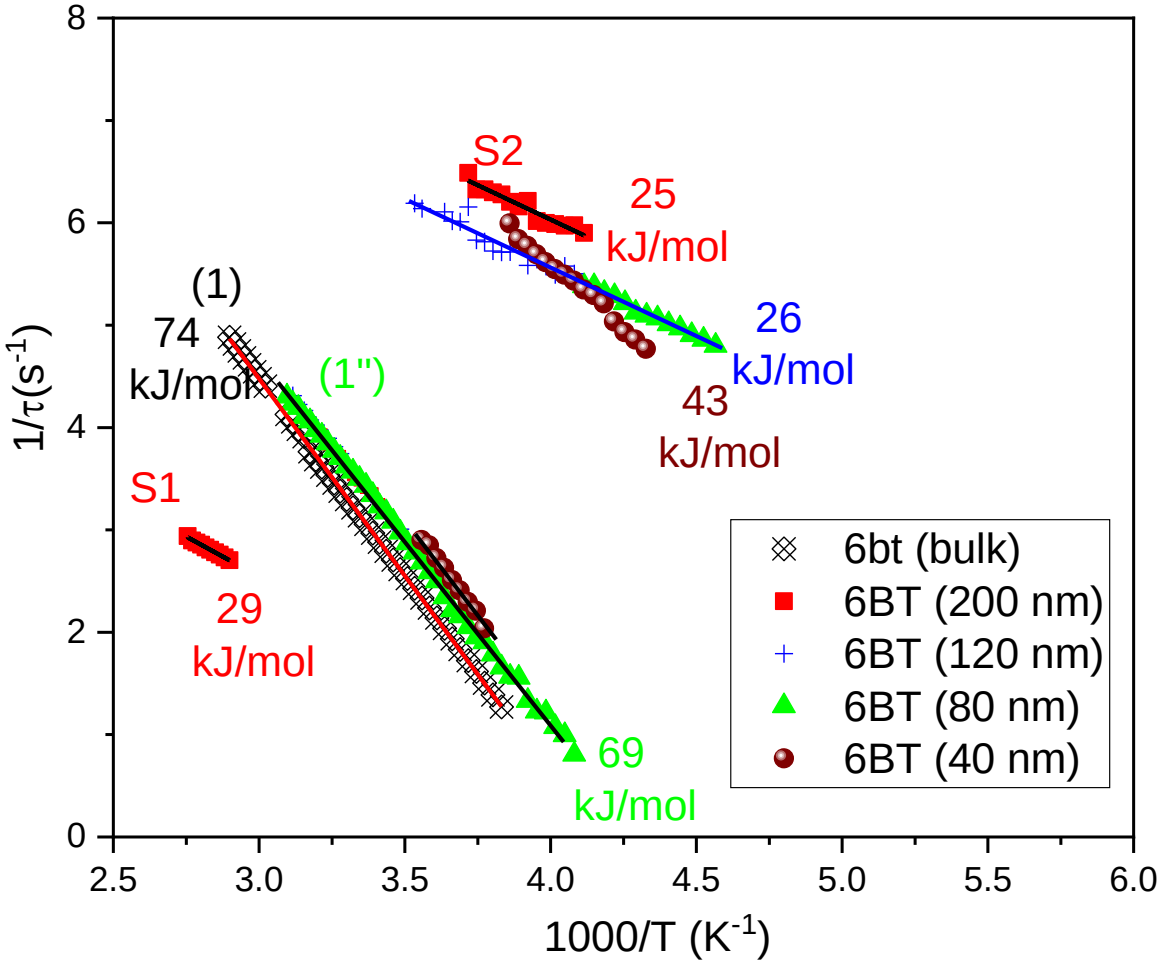
Comparison of the impact of „*hard*" and „*soft*" confinement on molecular dynamics of 6BT liquid crystal





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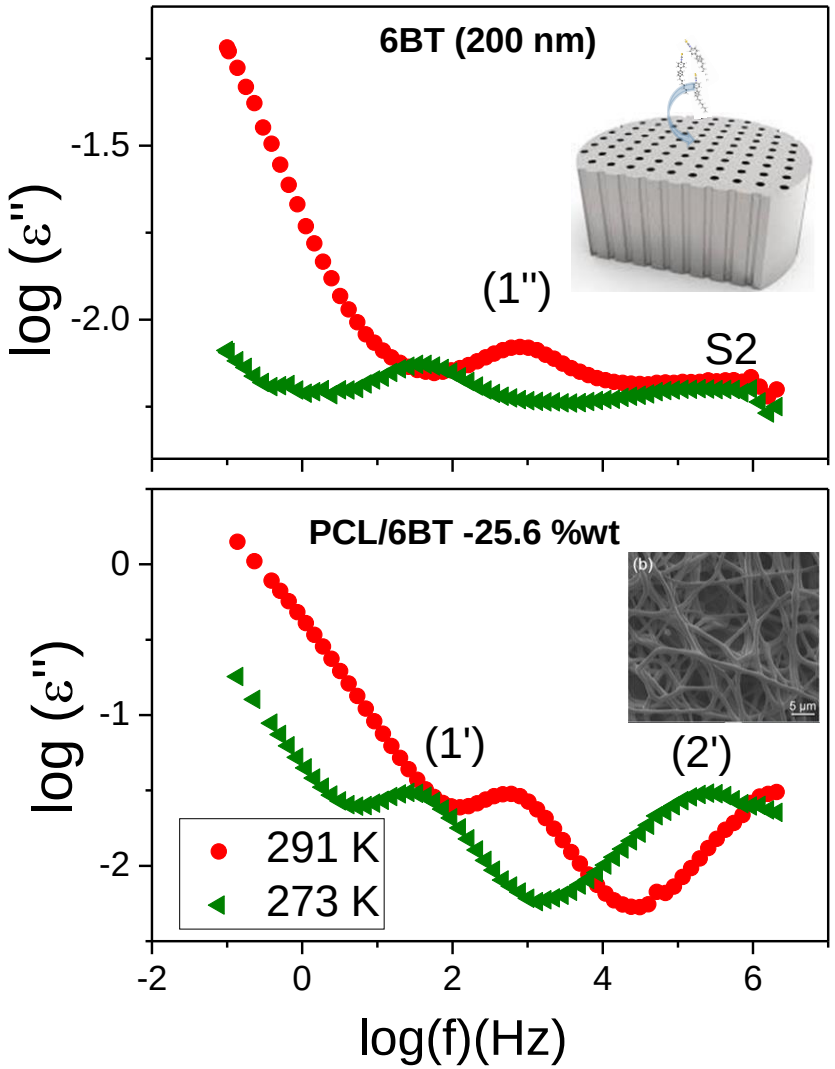

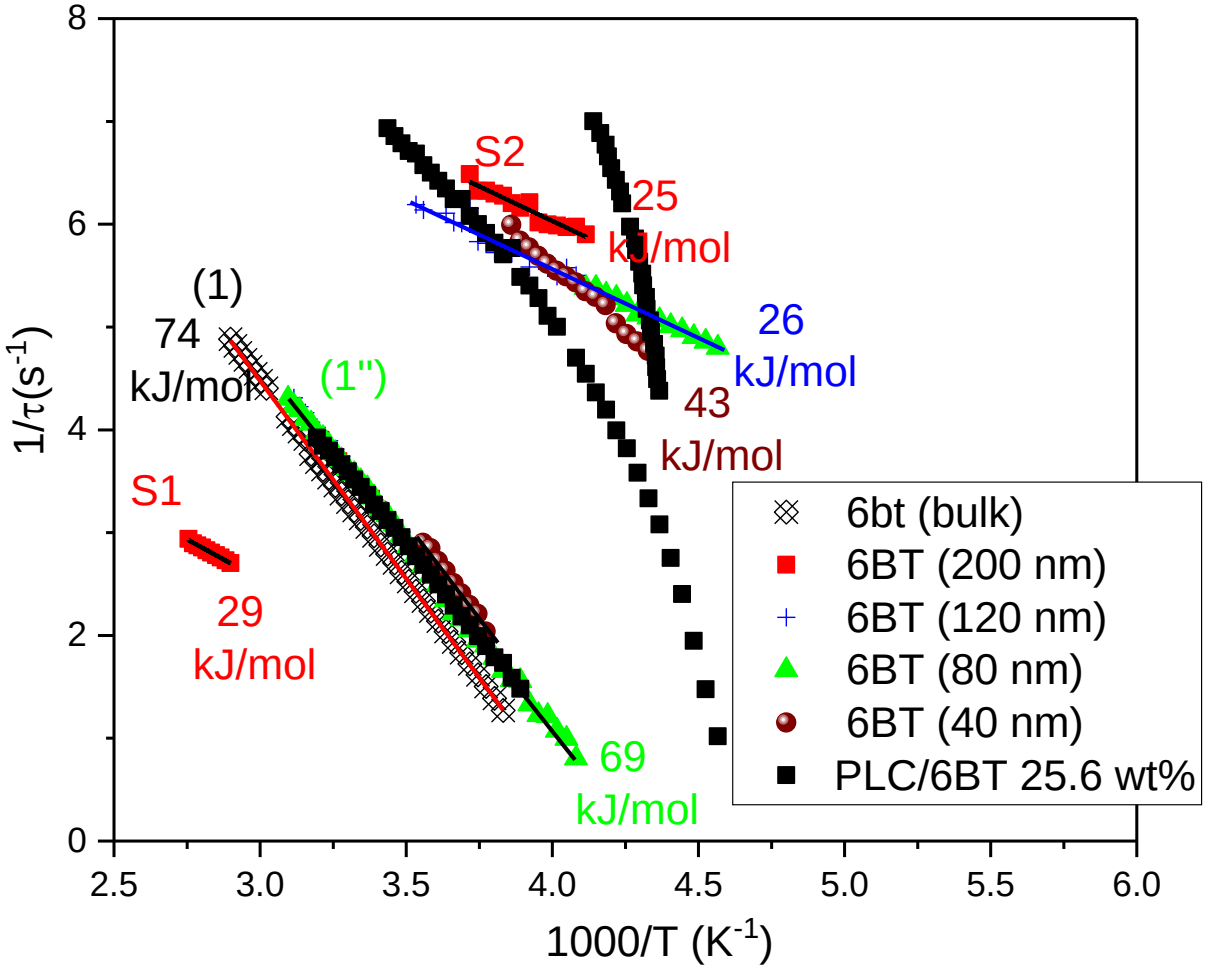

The rates of relaxation process around short axes of LC molecules in the composite fibers (25.6 wt % , 17wt% of LCs) coincide with those for 6BT in nanopores between 200 nm and 80 nm.

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How hard and soft confinement affect the molecular dynamics and crystallization process of LCs ?

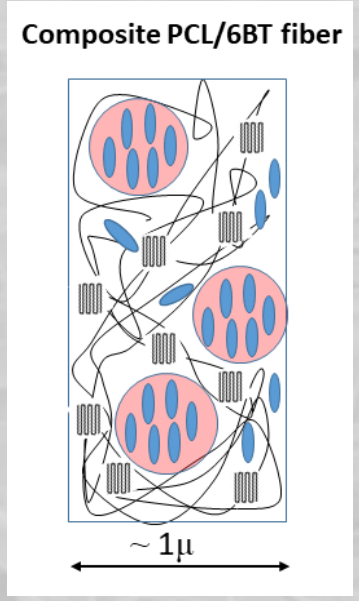
„*Hard*" confinement
6BT in nanoporous matrix
(SiO_2, AAO)

VS.

„Soft" confinement
LCs in polymer matrix

- Acceleration of motions of the *volume* molecules with respect to the bulk
- New relaxation processes
- Suppression of the crystallization with decreasing pores size

- **YES**
- **YES**
- **NO**



Conclusions

Behaviour of LCs in pores with diameter of several dozen nanometers

- The phase transition temperatures and the melting entropy have a linear decreasing relationship with the inverse pore diameter.
- Gradual course of crystallization in the pores is also reflected in the vibrational dynamics of alkyl chains
- The tendency for growth of a particular crystalline phases depends on the pore size and cooling rate.

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LC behaviour in pores of a few nanometer size

- the liquid crystal does not form highly-ordered smectic phases and surface interactions have a crucial influence on the arrangement of molecules in the pores.

Conclusions

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LC behaviour in pores of a few nanometer size

- the liquid crystal does not form highly-ordered smectic phases and surface interactions have a crucial influence on the arrangement of molecules in the pores.

Comparison of “hard” and “soft” confinement

- In contrast to “hard” confinemet, “soft” confinement enhances crystallization process
- The rotation of 6BT molecules around their short axes is accelerated in PCL/6BT composite fibres and the pores. In addition, mean relaxation times of this process in PCL/6BT fibres coincides with those obtained for the 6BT substance in large pores. These findings are also the first direct comparison of the influence of "soft" and "hard" spatial constraints on the relaxation processes of liquid crystal.

H1 **M. Jasiurkowska-Delaporte**, T. Rozwadowski, E. Dmochowska, E. Juszyńska-Gałązka, P. Kula, M. Massalska-Arodź, *Interplay between Crystallization and Glass Transition in Nematic Liquid Crystal 2,7-Bis(4-pentylphenyl)-9,9-diethyl-9H-fluorene*, Journal of Physical Chemistry B, **122** (2018) 10627; IF_{2018}=2.923

H2 **M. Jasiurkowska-Delaporte**, T. Rozwadowski, E. Juszyńska-Gałązka, J. Krawczyk, E. Dmochowska, P. Kula, M. Massalska-Arodź, *Relaxation dynamics and crystallization study in glass-forming chiral-nematic liquid crystal S,S-2,7-bis(4-pentylphenyl)-9,9-dimethylbutyl 9H-fluorene (5P-Am*FLAm*-P5)*, The European Physical Journal E, **42** (2019) 121; IF_{2018}= 1.686,

H3 **M. Jasiurkowska-Delaporte**, S. Napolitano, J. Ley, E. Juszyńska-Gałązka, M. Wübbenhorst, M. Massalska-Arodź, *Glass transition dynamics and crystallization kinetics in the smectic liquid crystal 4-n-butyloxybenzylidene-4'-n'-octylaniline (BBOA)*, Journal of Physical Chemistry B, **120** (2016) 12160; IF_{2016}=3.177

H4 **M. Jasiurkowska-Delaporte**, T. Rozwadowski, E. Juszyńska-Gałązka, *Kinetics of non-isothermal and isothermal crystallization in a liquid crystal with highly ordered smectic phase as reflected by differential scanning calorimetry, polarized optical microscopy and broadband dielectric spectroscopy*, Crystals, **9** (2019) 205; IF_{2019}= 2.061

H5 **M. Jasiurkowska-Delaporte**, *Isothermal and non-isothermal crystallization in liquid crystals as seen by broadband dielectric spectroscopy*, book chapter in "Advances in Dielectrics: Applications of Dielectric Spectroscopy to Crystallization Phenomena ", T. A. Ezquerra, A. Nogales (Ed.) Springer, (2020) ISBN 978-3-030-56185-7;

H6 **M. Jasiurkowska-Delaporte**, E. Juszyńska-Gałązka, P. M. Zieliński, M. Marzec, *Studies of molecular dynamics and non-isothermal crystallization process of 4-n-butyloxybenzylidene-4'-n'-octylaniline (BBOA) liquid crystal under two dimensional nano-confinement,* Journal of Molecular Liquids, **308** (2020) 113039; IF_{2019}=5.065,

H7 **M. Jasiurkowska**, W. Kossack, R. Ene, C. Iacob, W. Kipnusu, P. Papadopoulos, J. R. Sangoro, M. Massalska-Arodź, F. Kremer, *Molecular dynamics and morphology in confined 4-heptan-4'-isothiocyanatobiphenyl liquid crystals*, Soft Matter, **8**, (2012) 5194; IF_{2011}=4.561,

H8 **M. Jasiurkowska-Delaporte**, E. Juszyńska-Gałązka, W. Sas, P. M. Zieliński, A. Baranowska-Korczyc, *Soft versus hard confinement effects on the phase transitions, and intra- and inter- molecular dynamics of 6BT liquid crystal constrained in electrospun polymer fibers and in nanopores*, Journal of Molecular Liquids, **331** (2021) 115817; IF_{2019}=5.065

H9 **M. Jasiurkowska-Delaporte**, W. Kossack, W. K. Kipnusu, J. R. Sangoro, C. Iacob, F. Kremer, *Glassy dynamics of two poly(ethylene glycol) derivatives in the bulk and in nanometric confinement as reflected in its inter- and intra-molecular interactions*, Journal of Chemical Physics, 149 (2018) 064501; IF_{2018}=2.997

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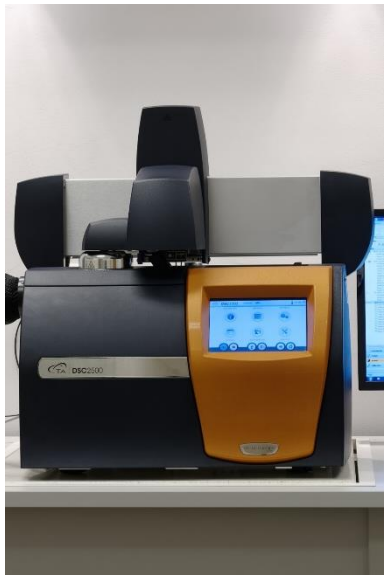

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Thank you for your attention